# LABORATORY MANUAL
ARRANGED TO ACCOMPANY THE
FOURTH EDITION OF
"A COURSE IN GENERAL CHEMISTRY"

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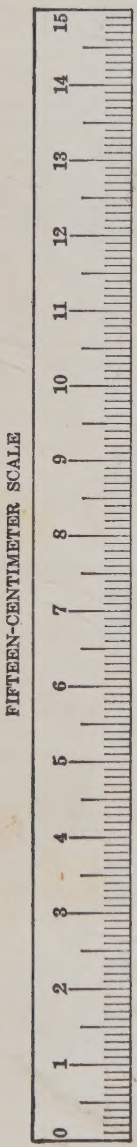
McPHERSON, HENDERSON, AND EVANS



FIFTEEN-CENTIMETER SCALE

# THE METRIC SYSTEM

**1. Length.** The unit is the *meter*. It is equal to 39.37 in.

10 millimeters (mm.) = 1 centimeter (cm.)
10 centimeters = 1 decimeter (dm.)
10 decimeters = 1 meter (m.)
1000 meters = 1 kilometer (km.)

The only measures of length ordinarily used by the chemist are the millimeter and the centimeter; thus, the average height of the barometer at the sea level is recorded as 76 cm. or, more commonly, as 760 mm., and not 7 dm. 6 cm.

**2. Volume.** The unit most in use is the *cubic centimeter*.

1000 cubic millimeters = 1 cubic centimeter (cc.)
1000 cubic centimeters = 1 cubic decimeter = 1 liter (l.)
1000 cubic decimeters = 1 cubic meter

The chemist uses only the cubic centimeter and the liter as measures of volume. Thus, the volume of a test tube is given as (say) 25 cc.; that of a flask as (say) 500 cc. or 0.5 liter.

**3. Weight.** The unit is the *gram*. This is the one thousandth part of the weight of a piece of platinum preserved at Sèvres, France. It is equal to 15.43 grains.

10 milligrams (mg.) = 1 centigram (cg.)
10 centigrams = 1 decigram (dg.)
10 decigrams = 1 gram (g.)
1000 grams = 1 kilogram (kg.)

The gram is practically the only unit used by the chemist. Thus, the weight of a crucible is given as (say) 10.532 g. and not 10,532 mg. or 10 g. 5 dg. 3 cg. 2 mg.

Note that

1 pound troy = 5760 grains = 373.24 grams
1 pound avoirdupois = 7000 grains = 453.59 grams
1 U.S. quart = 0.95 liter

Also note that

1 centimeter = nearly $\frac{2}{5}$ inch
1 kilogram = nearly $2\frac{1}{5}$ pounds (avoirdupois)

Eldon Winder
616 South Main
Ada Ohio

# LABORATORY MANUAL

## ARRANGED TO ACCOMPANY THE
## FOURTH EDITION OF
## "A COURSE IN GENERAL CHEMISTRY"

BY

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# PREFACE

The publication of the fourth edition of "A Course in General Chemistry" has made it necessary to revise the Laboratory Manual to accompany the text. The authors have gone over all the experiments with great care, discarding certain ones that were found to be too elementary or that educationally did not seem to repay the time required for them. During recent years many new experiments have been developed and tried out with large classes in our laboratory for first-year chemistry. Included in this list are "The Weight of a Definite Volume of Steam" and "The Molecular Formula of Steam" (Exercise 19); "The Quantitative Composition of Carbon Dioxide" (Exercise 26); "The Weight of a Definite Volume of Carbon Dioxide" and "The Molecular Formula of Carbon Dioxide" (Exercise 27). It is the belief of the authors that these experiments, as well as all others found in the Manual, will give satisfactory results with reasonable attention to detail and with the usual laboratory equipment.

It is not to be expected that any one student will perform all the experiments described. Students come from the secondary schools with a rather wide range of experience and attainments, and a laboratory instructor will desire opportunity to make selection of experiments suited to the attainments of individuals. In a number of exercises there are several quantitative experiments illustrative of much the same general principle. In such cases it is desirable to divide the class, making alternate assignments, so that all may profit from the variety in the exercises. The choice of experiments to be assigned or omitted must also depend upon the time available as well as upon the arrangement of continuance courses, if any.

The Appendix has been revised with care, to afford as much useful and helpful information as possible for the instructor and laboratory assistants. While the estimates for materials needed are based upon a class of ten students, it should be remembered that most chemicals are sold in a limited variety of standard packages and that purchases should be made in quantities that can be supplied from such stock.

The authors desire to express their great indebtedness to their associate, Dr. Jesse E. Day, who has spared no pains in suggesting and testing new experiments for the Manual.

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# GENERAL INFORMATION

A list of the apparatus and chemicals necessary for performing the experiments included in this Manual is given in the Appendix. It is expected that each desk will be provided with five of the most common reagents. (See Appendix for names and strength of each.) When any of these five reagents is mentioned in the list of materials required in performing each exercise, it will be understood that the reagents on the desk are the ones to be used unless otherwise specified. Other reagents used less frequently (see Appendix for details) should be placed on a reagent shelf available to all. Such reagents are designated in the exercises by the abbreviation "R.S." placed after the name of the reagent. The questions asked in each exercise are numbered consecutively. In the notebook, preface each answer with the number of the question.

The word *hood* signifies that the experiment is to be performed in a well-ventilated compartment, so that any poisonous gases evolved will be removed at once. All thermometer readings refer to the centigrade scale. The usual abbreviations for the metric system are used, such as, cc. for cubic centimeter, g. for grams, cg. for centigrams, mg. for milligrams, l. for liters.

It is assumed that every well-ordered laboratory has its own system of supplies, stock solutions, desk reagents, and locker inventory. Obviously, it will be necessary for the instructor to modify the directions given in the Manual to fit the arrangements of the laboratory.

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# LABORATORY MANUAL

## EXERCISE 1

## METHODS OF MEASURING LENGTHS, VOLUMES, AND WEIGHTS

### I. Measurement of the Lengths of Various Objects

**Object.** To measure the lengths of various objects on the centimeter scale and to acquire the ability to guess closely the length of an object on this scale.

**Apparatus.** 15-cm. scale (front inside cover); test tube; file; pencil; filter paper; square piece of window glass.

Note. Students unfamiliar with the metric system of weights and measures will find the necessary information and tables on the front inside cover of the Manual.

*a.* Using the scale on the front inside cover of the Manual, measure the length (in centimeters and fractions thereof) of various pieces of apparatus included in your outfit, such as a file, a test tube. Measure the diameter of your filter paper.

*b.* Estimate the length of your pencil and compare your guess with the length as measured on the scale. Similarly, estimate the lengths of the sides of your piece of window glass, and compare your guess with the measured lengths. Continue this practice until you can approximate closely by guess the length of a small object.

### II. Measurement of the Volumes of Liquids and the Capacities of Different Vessels

**Object.** To learn the methods used for measuring any desired volume of a liquid; also the methods used for finding the capacity of a vessel by determining the volume of a liquid it will hold.

**Apparatus.** 30-cc. graduated test tube; 250-cc. or 500-cc. graduated cylinder; beakers; test tube.

*a.* To measure any desired volume of a liquid, flasks of known capacities, or tubes or cylinders graduated in cubic centimeters (cc.), are generally used. A test tube graduated in cubic centimeters (Fig. 1) is very convenient for measuring small volumes of liquids, and a graduated cylinder serves for measuring larger volumes. In reading the volume of a liquid in a

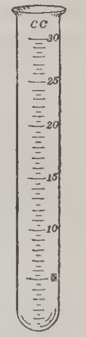

Fig. 1

graduated tube (or cylinder) always hold the tube so that your eye and the lower part of the *meniscus* (the curved surface of the liquid) are on the same horizontal plane; then read from the lower part of the meniscus (Fig. 2).

*b.* Pour water into the graduated test tube until it contains just 5 cc.; then transfer this to one of your ordinary test tubes and mark the lower part of the meniscus by a rubber band or a strip of paper cut from a label. Add 5 cc. more of water and mark the lower part of the meniscus as before. This will serve to fix in your mind the capacities of your test tubes and enable you to estimate the volumes 5 cc. and 10 cc. of a liquid.

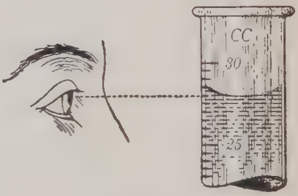

FIG. 2

Partly fill a test tube with water. Estimate the volume of the water, then pour the water into the graduated tube and see how nearly correct your estimate is. Continue in this way until you can approximate closely by guess any given volume of the liquid.

*c.* Fill one of your beakers with water to a point, say, 3 cm. from the rim of the beaker. Then pour the water into a graduated cylinder of suitable capacity, note the reading, and thus determine the capacity of the beaker when filled to the point designated. In a similar way determine the capacities of other beakers and flasks included in your outfit. Continue until you can approximate by guess the capacities of the different flasks and beakers in your outfit.

## III. Measurement of the Weights and Densities of Various Objects

**Object.** To learn the method of weighing different objects; also to learn the method of determining the densities of liquids and solids.

**Apparatus.** Platform balance; chemical balance; weights for balances; watch glass; small beaker; porcelain crucible; nickel five-cent piece; graduated test tube; burette.

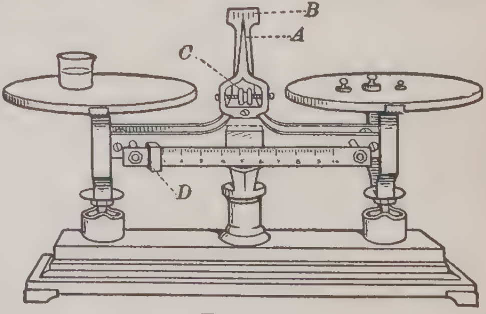

FIG. 3

**Materials.** Carbon tetrachloride (R.S.); rod of zinc; large iron nail; lump of sulfur.

*a. Balances and their uses.* The weights of different objects are determined by direct weighing. A number of different forms of balances are used by chemists. In a general way these fall into two classes: those which serve for weighing heavier objects up to 1000 g. with an accuracy of 0.5 g., and those which serve for weighing lighter

objects up to 50 or 100 g. with an accuracy of at least 0.01 g. For the former purpose an ordinary platform balance (Fig. 3) is convenient; for the latter purpose some one of the many forms of chemical balances (Fig. 4) may be used.

It is not advisable to attempt to give here complete, detailed directions in regard to the process of weighing; these will vary somewhat with the form of balance used and can best be furnished by the instructor in charge of the laboratory work. Nevertheless a few general statements in regard to the use of the different kinds of balances and the process of weighing will be helpful.

The chemical balance is a delicate instrument, and great care must be taken in using it. Such balances are provided with a thumbscrew (*D*, Fig. 4) by means of which the arms of the balance can be lifted from, or at least can be made to rest lightly on, the delicate edge upon which they swing. All changing of objects to be weighed, and also of weights, must be done when the pans are in this position. A turn of the knob *D* will then bring the arms into a position in which they are free to swing.

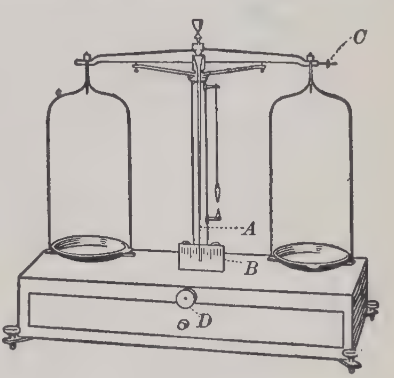

FIG. 4

Before using a balance the two arms should be in exact equilibrium. In the case of the platform balance, if the arms are in equilibrium, then the pointer *A* (Fig. 3) will point directly to the zero mark on the scale *B* when the empty pans are at rest (or if the pans are in motion, the pointer *A* will swing equal distances on the two sides of the zero point on the scale *B*); if it does not, bring it to this position by adjusting the screw *C*. In the more sensitive balance (Fig. 4) the pointer *A* rarely swings equal distances on the two sides of the zero point marked on the scale *B* when the pans swing. If the difference in the lengths of the swings is considerable, adjustment is made by turning the screw *C* in the proper direction.

For weighings not demanding great accuracy, approximate adjustment will suffice. For very accurate weighings, it is better to determine the true zero point on the scale rather than to attempt perfect adjustment of the balance. The method for doing this is described at the end of the exercise.

When the balance is adjusted, and the balance arms lifted, the object to be weighed is placed on one pan (the left is the more convenient) and weights (Fig. 5) are added to or removed from the other pan until the arms are in equilibrium as shown by the fact that the pointers *A*, *A* (Figs. 3 and 4) will swing equal distances on the two sides of the zero point on the scale. In the case of the platform balances the equivalent of 10 g. or any fraction of this weight is obtained by simply moving slide $D$ (Fig. 3) along the scale. When the arms are in equilibrium the weights are carefully noted and recorded. After a little practice one can by guess approximate closely the weight of the object and thus know about what weights must be added. *Care must be taken to read and record the weights accurately.* It is a good plan to check the readings of the weights by first noting the vacant places in the box in which the weights are kept and afterwards add up the weights as they are replaced in the box.

It must be remembered that a balance is a delicate instrument and must be given the same care as any other delicate instrument, such as a watch. The pans on which the weights and the object to be weighed are placed must be kept clean by means of a soft brush that will not scratch the pans. The objects to be weighed must be clean and dry, and the weights must be handled only with small forceps (Fig. 6). Under no condition must chemicals be placed directly on the scalepan. First weigh a suitable container, such as a watch glass or a small beaker; then place the material to be weighed in the container and again weigh.

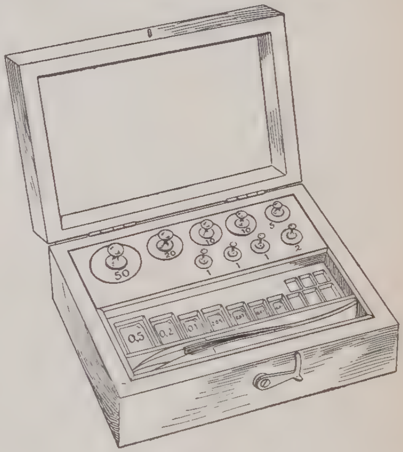

FIG. 5

After studying the construction of the balance and the method of using it, (1) weigh and record the weights of various small objects included in your outfit, such as a porcelain crucible or a watch glass. (2) What is the approximate weight of a nickel five-cent piece?

*b. Determination of the densities of liquids and solids.* The mass of a body is determined by direct weighing. The unit of mass is the gram, which, to a very close approximation, is equal to the weight of 1 cc. of water at its temperature of greatest density, namely, 4°. By the term *density* is meant the mass per unit volume. In terms of the metric system, *the density of any substance is the weight in grams of 1 cc. of that substance.* Since 1 cc. of water weighs 1 g., it follows that the density of any substance must equal its weight in grams divided by its volume in cubic centimeters, or

FIG. 6

$$D = \frac{M}{V}.$$

Determine the density of various liquids, such as water and carbon tetrachloride, by weighing in a small beaker a measured volume of each.

(3) Record your results. (4) Contrast your results with the densities of the same liquids as given in the text and account for any differences.

Determine the densities of various solids, such as a rod of zinc or aluminum or iron, or a lump of sulfur, as follows: First weigh the solid, recording the weight in grams. Then determine the volume of the solid by tying a thread to it and immersing it in a tube (graduated in cubic centimeters) partially filled with water. The graduated tube (Fig. 1) will serve for this purpose, but a burette (Fig. 63) is better, since its graduation is likely to be more accurate. The difference in the readings of the water in the tube before and after the immersion of the solid will give the volume of the solid. (5) Record your results in the following form:

DETERMINATION OF DENSITY OF ____________

| | |
|---|---|
| Weight of solid . . . . . . . . . . . . . . . . . . . . . | g. |
| Volume of solid . . . . . . . . . . . . . . . . . . . . . | cc. |
| Density of solid (Calculate) . . . . . . . . . . . . . . . | |
| Density given in text . . . . . . . . . . . . . . . . . . | |

*c.* (6) Suggest a method for determining the density of a solid (say, a lump of sugar) that is soluble in water.

*d. Accurate weighing by determining the zero point of the balance.* Even though a balance has been so perfectly adjusted that the pointer swings to equal distances on both sides of the zero point on the scale, it will not long remain so. For accurate weighings we determine the real zero point before each weighing, instead of attempting to readjust accurately the balance to the zero of the scale. Proceed as follows:

Make a record of an *unequal* number of consecutive swings of the pointer to left and to right, with no weights on the pans. Since the first and the last swings are on the same side of the zero point of the scale, the shortening of the swings due to friction is neutralized. The *average* swing for each side is calculated from your record, and the difference between these, divided by 2, is the true zero point of the balance. An example follows:

| | LEFT | | RIGHT |
|---|---|---|---|
| Swing (1) . . . . . . . . . . | 5.2 | Swing (2) . . . . . . . . . | 4.0 |
| Swing (3) . . . . . . . . . . | 5.0 | Swing (4) . . . . . . . . . . | 3.8 |
| Swing (5) . . . . . . . . . . | 4.8 | | |
| Average . . . . . . . . . | 5.0 | Average . . . . . . . . . | 3.9 |

5.0 (left) $- 3.9$ (right) $\div 2 = 0.55$ (left), the zero point for the balance.

In weighing, the weights must be adjusted until the pointer is swinging equally on both sides of the *true zero point* as just found.

## EXERCISE 2

## THE BUNSEN BURNER; MANIPULATION OF GLASS TUBING

**Object.** To study the Bunsen burner and to learn how to bend and otherwise handle glass tubing.

**Apparatus.** Bunsen burner as shown in Fig. 7; wing-top burner; hard-glass test tube with good cork to fit; set of cork borers; triangular file; round file; 500-cc. flask and cork to fit.

**Materials.** Wooden splint (1 cm. × 12 cm. is a convenient size); white cardboard 10 cm. square; soft-glass tubing 50 cm. in length; glass rod 25 cm. in length.

*a. The Bunsen burner.* The Bunsen burner, as employed in the laboratory, consists of the tube *A* (Fig. 7) screwed into the base *C*. The tube has two small openings near its lower part. A small band *B*, provided with similar openings, fits around the lower part of the tube in such a way that the openings of the tube may be closed or kept open by turning the band. Gas is admitted through *D* by means of rubber tubing.

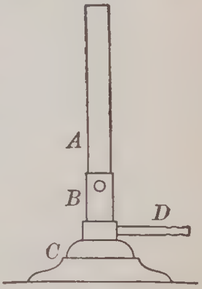

FIG. 7

Unscrew the tube and examine the different parts of the burner (Fig. 8); then put them together again and light the gas by holding a lighted match 4 or 5 cm. above the tube and turning on the gas. The gas supply should be adjusted so as to give a flame about 10 cm. high. The gas flowing through the tube mixes with the air drawn in through the openings in the lower part of the tube and burns with an almost nonluminous flame. If the band is adjusted so as to close the openings, the flame becomes luminous. Always use the nonluminous flame unless directed otherwise.

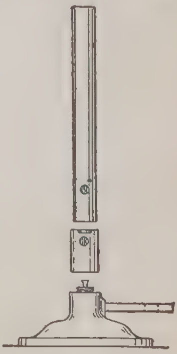

FIG. 8

(1) Hold a small wooden splint horizontally in the base of the Bunsen flame for two or three seconds and note the results. In the same way determine the relative temperatures of various parts of the flame. Turn the gas down until the flame is 7 or 8 cm. in height, then quickly thrust a piece of white cardboard, about 10 cm. square, *vertically* through the center of the flame, the lower end of the cardboard resting against the top of the burner. Remove the cardboard before it is ignited and from the scorched portions note the relative temperatures of different parts of the flame. (2) Draw a diagram to illustrate your results.

*b. To fit a tube with stopper and glass tube as shown in Fig. 9.* In all operations requiring the application of a strong heat to glass, the heat must be applied gently at first. Highly heated glass must be cooled slowly; otherwise it becomes brittle and easily broken.

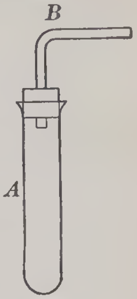

FIG. 9

From one of the lengths of soft-glass tubing cut a piece about 15 cm. in length. To do this, place the tubing on the desk and draw the edge of a triangular file across the point at which you wish to cut the glass. After the glass has been scratched, take the tube in the hands with the thumbs placed near together just back of the scratch (Fig. 10), and gently pull the glass apart, at the same time exerting a slight pressure with the thumbs. If the tube does not yield readily to a gentle pressure, a deeper scratch must be made. In the case of large tubing it may be found necessary to file a groove entirely around the glass. The edges of the cut tube will be sharp, and should be rounded by being rotated in the tip of the Bunsen flame (Fig. 11).

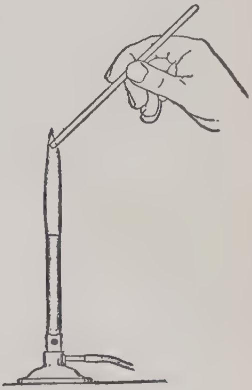

FIG. 11

To bend the glass tubing, first heat it, at the point where you wish to bend it, in the luminous Bunsen flame spread out by means of the *wing-top burner* (Fig. 12). Hold the tube lengthwise in the flame, gently rotating it so that all sides may be equally heated. Continue the heating until the glass bends easily, then remove it from the flame and quickly bend it to a right angle (*A*, Fig. 13). Great care must be taken to heat the tube uniformly to secure a rounded bend (*A*, Fig. 13); otherwise the bore of the tube will be contracted (*B*, Fig. 13), forming a bend which is not only unsightly but is easily broken.

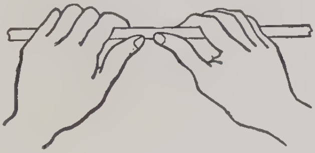

FIG. 10

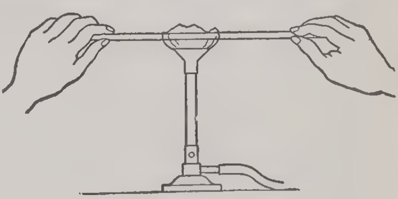

FIG. 12

Next select a cork of such a size that the smaller end will just enter the hard-glass test tube *A* (Fig. 9). Soften the cork by rolling it between the desk and a block of wood. Now insert into the cork the glass tube prepared as directed above. To do this, select a borer slightly smaller than the tube. Place the cork on the desk and cut half through it with the borer, not by punching but by rotating the borer under gentle pressure (Fig. 14); then reverse the cork and bore through from the other end. Care must be taken to keep the borer at a right angle to the top and base of the cork.

The glass tube, rounded at the edges, is now inserted in the cork by a gentle screwlike motion. If the hole is too small to admit the tube when a gentle pressure is applied, it may be slightly enlarged with a round file. Now put the cork in the test tube (Fig. 9) and set the apparatus aside for use in preparing oxygen (Exercise 6).

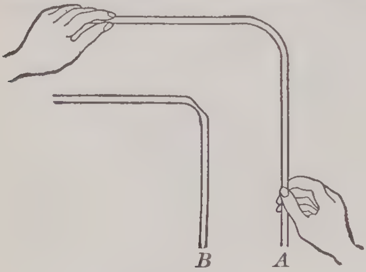

Fig. 13

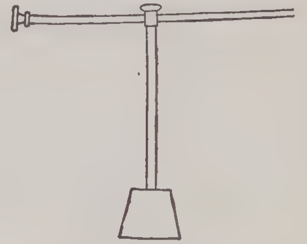

Fig. 14

*c. To make glass stirring rods.* Make two of them, one about 10 cm. and one about 15 cm. in length. Cut the rods to the proper length and round the ends by heating in the Bunsen flame (Fig. 11). Place the finished rods in the desk for future use.

*d. To make a wash bottle.* The student will often find a wash bottle useful in laboratory work. It should be made as represented in Fig. 15, the dimensions given in the figure being followed as closely as possible. A 500-cc. flask is used. *A* and *B* represent soft-glass tubes bent as shown in the figure. *B* is connected with the glass jet *D* by a piece of rubber tubing. The glass jet *D* is made as follows: Heat a piece of soft-glass tubing 12 or 15 cm. in length in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (*A*, Fig. 16). The tube must be constantly rotated, to prevent the softened portion from sagging. Now quickly remove the tube from the flame, and, holding

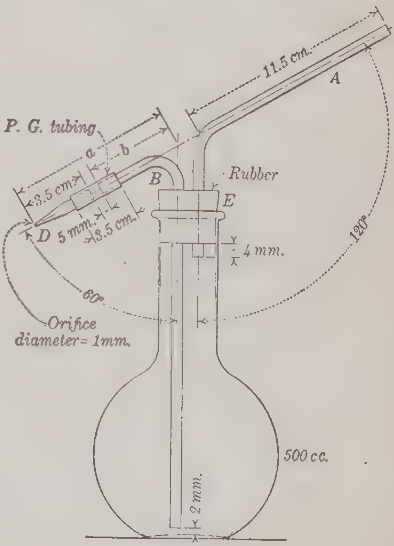

Fig. 15

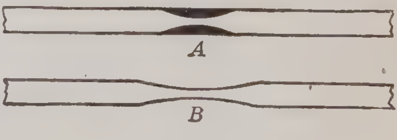

Fig. 16

it in a vertical position, gently pull the ends apart until the bore is of the desired size (*B*, Fig. 16). The glass jet is then formed by cutting the tube at *B* and rounding the edges in a flame.

## EXERCISE 3

## SOME PRELIMINARY MANIPULATIONS

**Object.** To learn various manipulations, such as the proper method of heating liquids in test tubes, of pouring liquids from one container into another, and of separating a solid from a liquid by filtration.

**Apparatus.** Test tubes; glass rod; 2 beakers; funnel and filter paper; ring stand and burner; evaporating dish.

**Materials.** Piece of chalk; 2 or 3 g. of common salt.

*a. Heating liquids in test tubes.* Half fill a test tube with water and apply heat until the water boils rapidly. To do this, hold the test tube between the thumb and fingers (Fig. 17), constantly rotating it on its axis so as to apply the heat uniformly. The heat should be applied to the upper portion of the liquid, care being taken, however, that the flame does not strike the tube above the level of the liquid. In case the upper part of the tube becomes heated, it may be supported by a test-tube holder (Fig. 18) or by a band of paper wrapped about the upper part of the tube (Fig. 19). The sudden formation of vapor sometimes causes the contents of the tube to be thrown out; hence *care must be taken not to point the tube toward anyone* (Fig. 20).

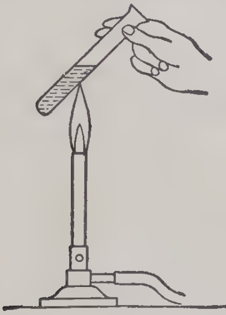

FIG. 17

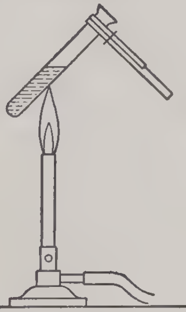

FIG. 18

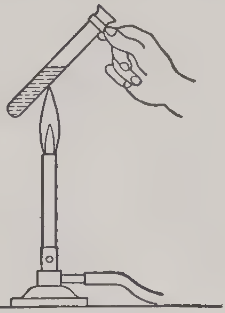

FIG. 19

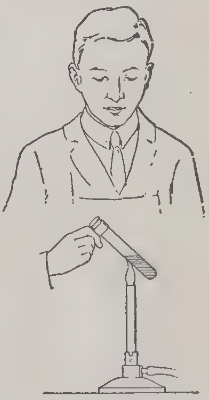

FIG. 20

*b. Pouring liquids from one vessel into another.* In doing this, care must be taken to prevent the liquid from running down the side of the vessel from which it is poured. A glass rod should be held lightly against the rim of the vessel, as shown in Fig. 24. The liquid will then flow down the rod instead of following the surface of the vessel.

Fill a beaker with water and transfer it slowly to another vessel without using the glass rod; repeat, using the glass rod.

In pouring liquids from bottles a glass rod may be used, or the neck of the bottle may be placed lightly against the rim of the vessel into which the liquid is being poured (Fig. 21). This will prevent the liquid from running down the side of the bottle. The stopper must never be laid down on the desk. Catch it between the fingers, as shown in Fig. 22. This leaves the hand free to grasp the bottle, as shown in Fig. 21.

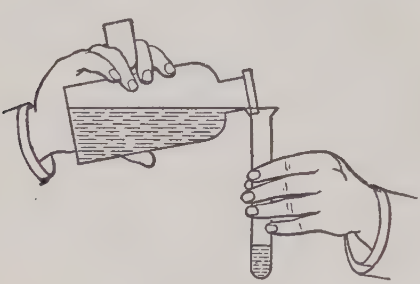

Fig. 21

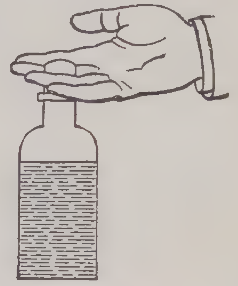

Fig. 22

*c. Decantation; filtration; solution; evaporation.* It is often necessary to separate a liquid from a finely divided solid which is suspended in it. This may be done by one of the two following methods:

1. *Decantation.* When the solid is heavy and readily settles to the bottom of the vessel, the liquid may be carefully poured off, or *decanted.* Thus, sand suspended in water may be separated from the liquid in this way.

2. *Filtration.* Usually, however, the solid will not readily settle or will do so only after long standing. In such cases the mixture is *filtered*, that is, poured on a filter paper, which allows the liquid to run through but retains the solid. To prepare the filter paper, fold the paper *A* (Fig. 23) along a diameter into halves, *B*, then, at right angles to the first fold, into quarters, *C*. The folded filter is then opened so as to form a cone, *D*, half of which is composed of three thicknesses of paper and the remainder of one thickness. Now fit the cone into a funnel *E* of such a size that the paper does not quite reach the top. The paper must accurately fit the funnel; if it does not, make it do so by varying the angle of the fold. Place the paper in the funnel and thoroughly wet it with water. After the water has run through, press

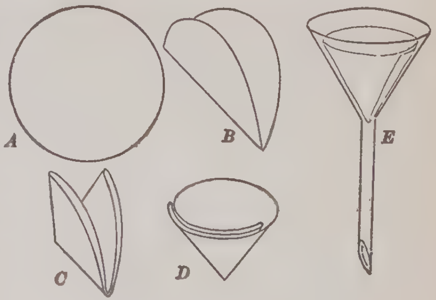

Fig. 23

the paper firmly against the sides of the funnel with the finger so as to remove any air bubbles. The filter is now ready for use (Fig. 24).

*Solution.* The process of filtration not only enables us to separate liquids from solids but also certain solids from each other. To illustrate this, grind a piece of chalk to a powder in a mortar, and mix the product intimately with about an equal bulk of common salt. Place the mixture in a small beaker, add about 50 cc. of distilled water, and stir with a glass rod. (The ends of the rod should be rounded by rotating them in a flame; otherwise the beaker may be scratched and easily broken.) The salt dissolves, forming a *solution.* Filter off the insoluble chalk, collecting the *filtrate,* that is, the clear liquid which passes through the filter paper, in a beaker (Fig. 24).

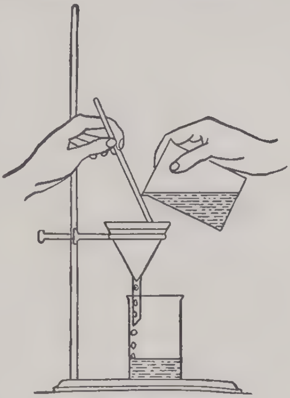

FIG. 24

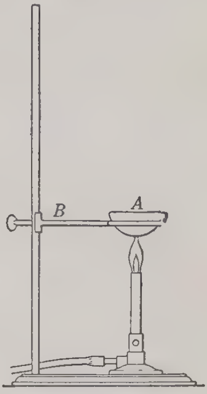

FIG. 25

*Evaporation.* The salt may be recovered from the filtrate by the process of *evaporation.* To perform this operation, pour the filtrate into an evaporating dish *A* (Fig. 25), support the dish on a ring stand *B*, and heat gently. The liquid may be made to simmer, but should not actually boil. Withdraw the heat as soon as the water is evaporated. Note the residue left. Convince yourself that it is common salt.

## EXERCISE 4

### CHEMICAL ACTION; VARIETIES OF MATTER

**Object.** To study the characteristics of chemical action; also to learn the distinction between elements, compounds, and mixtures.

**Apparatus.** Burner; evaporating dish; ring stand; magnet; magnifying glass; test tubes.

**Materials.** 10 cm. of glass tubing; wooden splint; 2 or 3 g. of sugar; 1 g. of common salt; piece of mossy zinc; sulfuric acid; 2 g. of sulfur; 2 g. of powdered iron.

*a.* Select two short pieces of glass tubing and heat one of these (gently at first) in the Bunsen flame until it is red hot; then lay it aside to cool. When it is cold, compare it with the piece that was not heated. (1) So far as you are able to judge, has the heat changed the properties of the glass in any way? Suppose you were to break the glass into pieces, (2) would the characteristics of the glass be thereby changed? Such changes as these which take place when glass is heated or broken into pieces, and which do not in any way change the composition of the matter, are known as *physical changes.*

*b.* Repeat *a*, using a splint of wood in place of the glass tube. (3) How does the change that occurs differ from that in *a*? (4) What term is applied to such changes? (5) Define the term.

*c.* Place enough sugar in a clean, dry test tube to cover the bottom to a depth of 1 cm. Heat it gently in the tip of the flame *as long as any changes are produced.* Then cool the residue. Taste it and try its solubility in water. (6) Contrast the properties of the sugar with the residue left on heating it. (7) Do any properties remain unchanged? (8) What grounds have you for assuming that a chemical action has taken place?

*d.* Place about 1 g. of common salt in a test tube and dissolve it in a little water. Pour the clear solution into an evaporating dish and evaporate to dryness (Fig. 25). (9) What is the residue, and what are your reasons for your conclusion?

*e.* Cover a small piece of zinc in a test tube with about 5 cc. of water and add *carefully* 6 or 8 drops of sulfuric acid, mixing the contents after the addition of each drop. Notice that the zinc dissolves with the evolution of a gas. Hold a burning splint at the mouth of the test tube and (10) note the result. After the action has entirely ceased, filter off any undissolved zinc and evaporate the solution to dryness (***Hood***) as in *d.* (11) How does the change differ from that in *d*?

*f. Elements; compounds; mixtures.* (12) Define the term *element.* (13) Are iron and sulfur included in the list of elements? Weigh out separately (on paper) 2 g. of sulfur and 2 g. of powdered iron and (14) make a careful list of their properties, including color, odor, solubility in water, response to magnetic attraction. Now mix the two, and grind them together intimately in a mortar. Examine the product with a magnifying glass. (15) Can you distinguish the iron from the sulfur? (16) Can you separate them with a magnet? (17) Have they undergone any change in properties? (18) What is the product of mixing called?

Now place the material in a clean test tube and heat gently. As soon as the mass begins to glow, quickly withdraw the tube from the flame. Note that the mass continues to glow. Now heat it strongly for one or two minutes; then cool the tube, remove the substance (it may be necessary to break the tube to do so), and examine the product with a magnifying glass. (19) Can you now distinguish between the iron and the sulfur? (20) Try the effect of the magnet. (21) Of what is the product composed? (22) When elements combine chemically, do they retain their original properties? (23) What is the product of such a combination called?

*g.* (24) How do you tell whether or not a chemical reaction is involved in any given change? (25) Point out four chemical reactions brought about in performing the experiments in Exercise 4. (26) From the energy standpoint define the terms *compound* and *mixture.*

## EXERCISE 5

### COLLECTION OF GASES; PREPARATION OF OXYGEN

**Object.** To learn how to collect gases; also how to prepare oxygen.

**Apparatus.** 250-cc. wide-mouthed bottles; glass plate to cover mouth of bottle; 50 cm. (or more) of rubber tubing; pneumatic trough or graniteware pan; ring stand and clamp; wing-top burner; hard-glass test tube; apparatus as shown in Fig. 28.

**Materials.** 1 g. of mercuric oxide; wooden splint; 5 g. of sodium peroxide.

*a. Collection of gases.* Fill a wide-mouthed bottle (250-cc.) with water. Cover its mouth with a glass plate, being careful to exclude all air bubbles. Hold the plate firmly in place, invert the bottle, and dip its mouth into the water in a pneumatic trough. Remove the glass plate. (1) Why does the water remain in the bottle? Now fill the bottle with exhaled air by placing one end of a piece of glass or rubber tubing under the mouth of the bottle and blowing gently through the other end. Before the bottle, so filled, is removed from the trough, cover its mouth tightly with a glass plate. The bottle so covered may then be placed on the desk in either an upright or an inverted position. (2) When should it be placed in an inverted position?

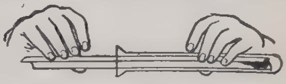

FIG. 26

Fill a bottle with exhaled air and then transfer the air to another bottle. (3) Draw a diagram to show the method of doing this.

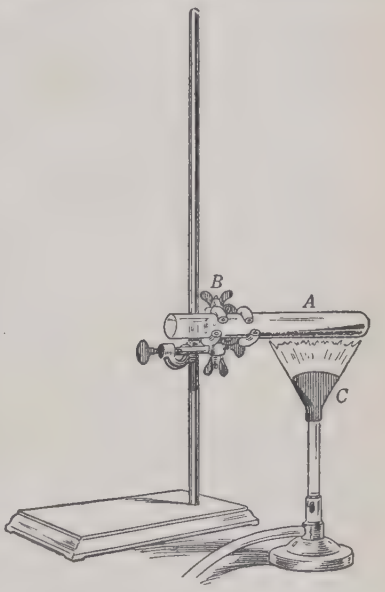

FIG. 27

*b. Preparation of oxygen from mercuric oxide.* In the bottom of a clean, dry hard-glass test tube place about 1 g. of mercuric oxide. This is done by placing the oxide near the end of a narrow strip of folded paper and introducing it carefully into the tube, as shown in Fig. 26. On inclining the tube and gently tapping the paper the oxide will be deposited in the bottom of the tube. The paper is now withdrawn, leaving the sides of the tube perfectly clean. By gently tapping the tube while rotating it between the fingers, the oxide may be spread out in a layer along the bottom half of the tube. Support the test tube *A* in a horizontal position by means of a ring stand and clamp *B* (Fig. 27), being careful to clamp the test tube gently so as to avoid risk of breaking it. Heat the mercuric oxide gently with a flame spread out by a wing-top burner *C*.

During the heating insert a glowing splint from time to time into the mouth of the tube. (4) Note the result. Continue to heat as long as any gas is evolved. (5) What remains in the tube? (6) How has the heat affected the mercuric oxide? (7) What kind of change has the mercuric oxide undergone? (8) State the class of substances (element, compound, or mixture) to which mercuric oxide belongs, and give reasons for your answer.

*c. Preparation of oxygen from sodium peroxide.* Sodium peroxide is a white solid compound of sodium and oxygen containing 41 per cent of oxygen. When treated with water, a part of this oxygen is set free. To prepare oxygen by this method arrange an apparatus according to Fig. 28. By means of a short piece of rubber tubing *A* connect the funnel *B* with a glass tube *C*, pinching the rubber tube *A* shut with a screw clamp. Place about 5 g. of sodium peroxide in the bottom of *D* and partly fill the funnel with warm water. Very cautiously open the screw clamp so that the water will run down and fall, drop by drop, on the peroxide. The funnel must be kept partly filled with water so that no air will be drawn into the bottle *D*. A steady current of oxygen is given off. Allow some of the gas to escape through the water; then collect a bottle of the gas and test it by thrusting a glowing splint into the bottle. (9) What is the source of the oxygen? (10) What substances remain in the generator bottle (page 31 of text)? (11) Why allow some of the gas to escape before beginning to collect the oxygen?

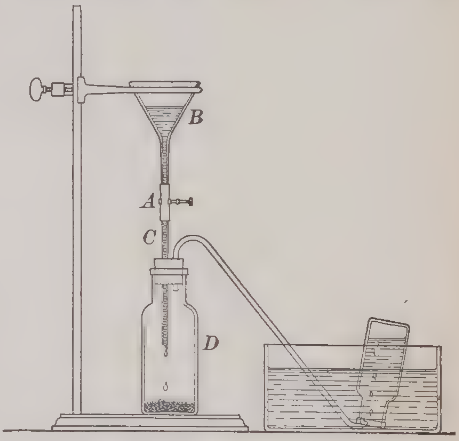

FIG. 28

## EXERCISE 6

### OXYGEN

**Object.** To learn how oxygen is ordinarily prepared in the laboratory and to study the properties of the gas.

**Apparatus.** Test tubes; test tube prepared in Exercise 2 (Fig. 9), connected with rubber tube as shown in Fig. 29; trough and wide-mouthed bottles shown in Fig. 29; burner; deflagrating spoon; 4 pieces of window glass 10 cm. square; funnel and filter paper; evaporating dish; ring stand.

**Materials.** 10 g. of potassium chlorate; 4 g. of manganese dioxide; wooden splint; 1 g. of sulfur; picture-frame wire 20 cm. long; bit of cotton.

*a. Preparation of oxygen from potassium chlorate (preliminary experiment on catalysis).* Select two test tubes of the same size, and clean and dry them thoroughly. Into the one introduce 2 g. of potassium chlorate (Fig. 26); into the other introduce 2 g. of potassium chlorate mixed intimately with 1 g. of manganese dioxide.

Now heat the contents of the two tubes, applying the flame so that both tubes, so far as possible, are equally heated. Repeatedly thrust a glowing splint into each tube in order to detect any oxygen that may be evolved. (1) Note the results. (2) What effect has the manganese dioxide? (3) Does the addition of the manganese dioxide enable one to obtain an increased amount of oxygen (page 33 of text)? (4) What term is applied to substances that act like the manganese dioxide?

*b. Preparation of oxygen (usual laboratory method).* Mix intimately on paper 6 g. of potassium chlorate and 3 g. of manganese dioxide. The presence of impurities in the materials may lead to a serious explosion when heat is applied; hence test a small portion of the mixture, say 0.5 g., by heating it in a test tube. In the absence of impurities the oxygen is evolved quietly, unaccompanied by any very marked sparking in the materials.

Place the apparatus prepared in Exercise 2 (Fig. 9) on a ring stand as shown in Fig. 29, and connect with it a piece of rubber tubing *C*; also fill four wide-mouthed bottles (250-cc.) with water and invert them in a pneumatic trough, as shown in the figure. Now transfer the mixture of potassium chlorate and manganese dioxide to the glass tube *A* (Fig. 29) and insert the cork; then, holding the burner in the hand, heat the mixture gently with a small flame, applying the heat at first to the upper part of the mixture. The flame must not strike the upper part of the test tube, as the cork may be charred or ignited. At first the heat expands the air and a few bubbles escape; then the oxygen is evolved. Regulate the heat so as to secure a uniform and not too rapid evolution of the gas. (5) Will the gas which passes over at first be the pure oxygen? Collect four 250-cc. wide-mouthed bottles of the gas. Before the heat is withdrawn, remove the cork from the tube. (6) Why? (7) What is the source of the oxygen? Put the tube and contents aside for use as described in *e*.

*c. Properties of oxygen.* (8) Note the physical properties (color, odor, taste, solubility in water) of the gas. (The slight cloud that is often present when oxygen is prepared from potassium chlorate is due to an impurity and will disappear if the gas is allowed to stand over water.)

*d. Chemical conduct of oxygen.* Repeatedly thrust a glowing splint into a bottle of the gas. (9) Account for the results.

Heat some sulfur in a deflagrating spoon until it begins to burn. (10) Note the color and size of the sulfur flame; then lower the burning sulfur into a bottle of oxygen and note the change. (11) What name can you give to the product formed (page 37 of text)?

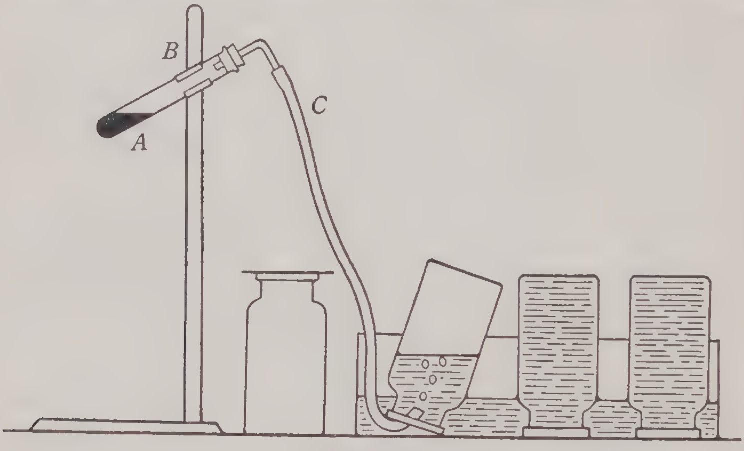

FIG. 29

Tip the piece of picture-frame wire with sulfur by wrapping a very small bit of cotton about the end of the wire and dipping this into melted sulfur (for this purpose melt a little sulfur in a deflagrating spoon). Ignite the sulfur by holding it in a Bunsen flame for an instant, and then thrust the wire into a bottle of oxygen. (12) Describe the results and account for the change. (13) What is the product formed? (14) Write a brief summary of the properties and chemical conduct of oxygen, based on the above experiments.

*e. Separation of the compounds present in the residue left in the preparation of oxygen.* Heat the tube *A* (Fig. 29) containing the residue until no more oxygen is evolved. After the tube is cool, nearly fill it with water and shake the contents thoroughly. After a few minutes filter off the solid matter ((15) What is it?) and collect the filtrate in a small beaker. Repeat the filtration if necessary, in order to obtain a perfectly clear filtrate. Set the beaker containing the filtrate on your desk for a

day or two, until most or all of the water has evaporated and crystals are deposited. Convince yourself that the crystalline substance is different from the potassium chlorate with which you started. (16) What is the compound (page 33 of text)? $MNO_2$

## EXERCISE 7

### COMBUSTION

**Object.** To determine the changes that take place when a substance burns.

**Apparatus.** Porcelain crucible; burner; ring stand; pipestem triangle to support the crucible while being heated; iron wire 10 or 12 cm. long; pneumatic trough (or dish); wide-mouthed bottle or large beaker; candle.

**Materials.** 2 or 3 g. of powdered iron (use iron reduced by alcohol); 2 or 3 g. of mossy tin; pellet of phosphorus, size of a small pea (to be obtained from the instructor when needed); 5 cc. of limewater (R.S.).

*a. The burning of metals.* Place 2 or 3 g. of powdered iron in a porcelain crucible and accurately weigh to 0.01 g., (1) recording the weight. Now heat the crucible (Fig. 30) until the iron begins to glow (burn); then withdraw the heat. (2) Does the iron continue to burn? After the crucible is cool, reweigh. (3) Compare the weight of the product with that of the unburned iron and account for any difference there may be. The experiment may be repeated, using tin.

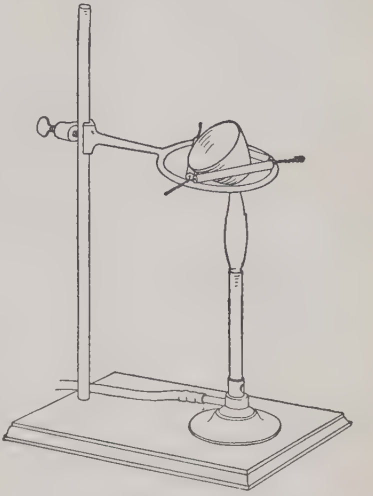

FIG. 30

***Caution.*** *Phosphorus must be kept and handled only under water; otherwise it may ignite and cause serious damage.*

*b. The burning of phosphorus.* Cover the bottom of a pneumatic trough with water to a depth of 2 or 3 cm. In the water set a porcelain crucible containing a piece of phosphorus the size of a small pea.

Ignite the phosphorus by touching it with a hot wire or the end of a hot file, and quickly invert over the crucible a wide-mouthed bottle (or beaker) (Fig. 31), being careful to keep the mouth of the bottle below the surface of the water. The white powder formed by the burning phosphorus floats in the air in the bottle but is gradually dissolved by the water.

Leave the bottle in position until the powder has entirely disappeared. Note that the water has risen in the bottle. (4) How do you account for this fact? (5) Show that your conclusion is in accord with the results obtained by burning iron in air. Suppose it were possible for you to collect and weigh the white powder formed by the burning phosphorus, (6) how should you expect its weight to compare with that of the unburned phosphorus?

*c. The burning of a candle (preliminary experiment).* Pour 5 cc. of clear limewater into a small beaker and by means of a glass tube gently blow exhaled air through the liquid. Note that the liquid soon becomes cloudy, or milky, in appearance. This change is due to the action of the colorless and odorless gas known as *carbon dioxide* (which is present in the exhaled air) and constitutes a good test for this gas.

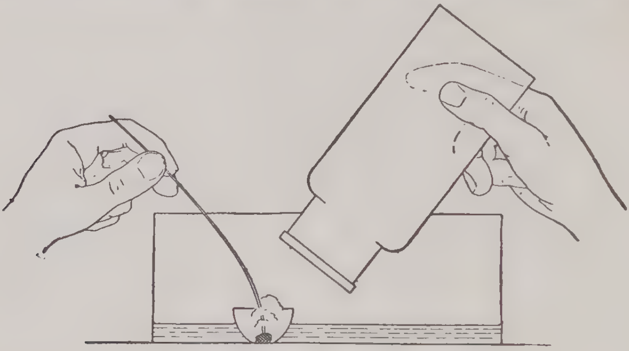

FIG. 31

Hold a cold, dry wide-mouthed bottle over a candle flame, as shown in Fig. 32. Note the film of moisture collecting on the bottle. After one or two minutes remove the bottle quickly and pour into it 5 cc. of clear limewater. Place the palm of the hand tightly over the mouth of the bottle and shake the contents. (7) Note any change in the appearance of the limewater. (8) What does the change prove? (9) What are the products of the combustion of the candle? (10) Account for their formation (page 38 of text). (11) How do you account for the fact that a burning candle gradually loses weight, while iron increases in weight on burning? There is a small percentage of carbon dioxide in the air. (12) Show by experiment that the change in the appearance of the limewater is not due to the carbon dioxide in the air.

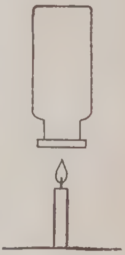

FIG. 32

*d.* (13) Define the terms *oxide, oxidation, product of combustion,* and *kindling temperature,* and give an illustration of each taken from the above experiments (Exercise 7).

## EXERCISE 8

## HYDROGEN

**Object.** To prepare hydrogen and to study its properties.

**Apparatus.** Test tubes, forceps, and trough, as shown in Fig. 33; bottles (250-cc., wide-mouthed) and apparatus as shown in Fig. 34; beaker; stirring rod; 60-cc. bottle; ring stand and burner; evaporating dish; funnel and filter paper.

**Materials.** Two pieces of sodium (size of a small pea); few drops of kerosene; wooden splints; 1 g. each of bits of iron, aluminum, magnesium, and copper; 10 g. of mossy zinc; copper sulfate solution (R.S.); dilute hydrochloric acid prepared by adding 15 cc. of the concentrated acid to 15 cc. of water; dilute sulfuric acid prepared as directed below.

Preparation of dilute sulfuric acid: *Great care must be taken in this preparation,* for heat is generated when concentrated sulfuric acid and water are mixed. If care is not taken, so much heat may be generated as to cause the liquid to boil and spatter, and this hot liquid, if splashed onto any sensitive part of the body, will cause serious injury. To prepare the dilute acid proceed as follows: Pour *slowly*, a few drops at a time, 20 cc. of the concentrated sulfuric acid into a beaker containing 65 cc. of water. Stir the water constantly with a glass rod while the acid is being added. *Notice that the acid is poured into the water — never the reverse.* Cool the mixture by placing the beaker in cold water.

*a. Preparation from water.* Fill a test tube with water and invert it in a beaker of water. Wrap a piece of sodium in a bit of filter paper previously moistened with kerosene. Raise the inverted test tube until its mouth dips just below the surface of the water in the beaker, grasp the sodium, wrapped in the paper, with forceps and (Fig. 33) quickly bring it into the mouth of the test tube and release it. The sodium, being lighter than water, will rise to the top of the tube. Stand at arm's length, as a slight explosion sometimes occurs. Notice that the sodium decomposes the water, liberating a gas which is caught in the tube. When the action has ceased, place your thumb tightly over the mouth of the tube to prevent the gas from escaping, and bring the tube to an upright position. Light a splint, remove the thumb from the tube, and quickly bring the flame to the mouth of the tube. (1) Does

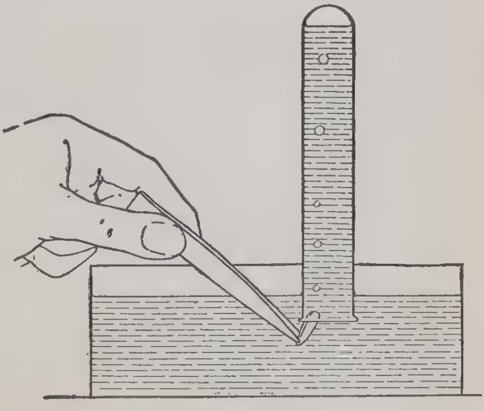

FIG. 33

the gas burn? (2) What is the source of the gas? (3) What compound is formed in the reaction (page 51 of text)? (4) What other methods may be employed for obtaining hydrogen from water?

*b. Preparation from acids (preliminary experiment).* All acids contain hydrogen; when certain metals (those above hydrogen in the electrochemical series, page 298 of text) are brought in contact with acids, the metal takes the place of the hydrogen in the acid. The hydrogen is thus set free and may be collected over water.

Place five test tubes in your test-tube rack. In the bottom of the first, place a few pieces of iron (tacks); in the second, a bit of sodium; in the bottoms of the third, fourth, and fifth, respectively, place some thin pieces (in the form of shavings or ribbons) of the metals aluminum, magnesium, and copper. Now add to each tube 3 or 4 cc. of the dilute hydrochloric acid, a few drops at a time, so that the action may not become too violent. (5) Is a gas (hydrogen) evolved in each case? If the acid acts on the metals with different rapidity, (6) note the order of this action. (7) Is the gas combustible (test by bringing a lighted splint to the mouth of the tube)? (8) What elements are present in each of the compounds formed (page 52 of text)? The compounds formed are solids which remain dissolved in the water present. (9) How could you recover them?

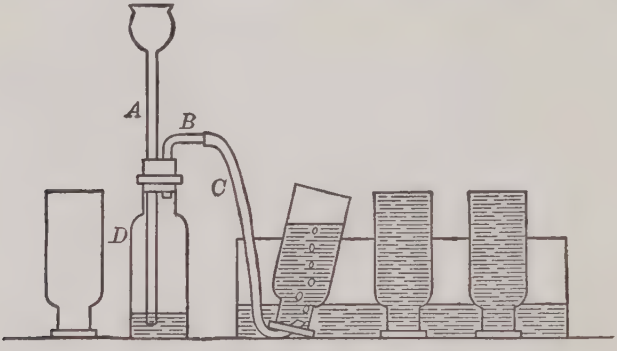

FIG. 34

*c. Preparation from acids (usual laboratory method).* While many different acids and metals may be used in the preparation of hydrogen, for laboratory purposes it has been found most convenient to use zinc and dilute sulfuric acid. To prepare hydrogen in this way, proceed as follows:

Prepare a hydrogen generator according to Fig. 34. *D* represents a wide-mouthed bottle of about 250-cc. capacity. The gas-delivery tube *B, C* is the same as that used in the preparation of oxygen (Fig. 29). A rubber stopper for the bottle *D* is preferable, although a good cork stopper will do. The funnel tube *A* must extend nearly to the bottom

of the bottle. (10) Why? Put about 10 g. of mossy zinc ((11) Why so named?) into *D* and add to the zinc 8 or 10 drops of a solution of copper sulfate. Now stopper the bottle as shown in Fig. 34 and pour just enough water through the funnel tube to cover the zinc.

The apparatus *must be air-tight.* Prove that it is so by blowing into the delivery tube *C* until the water is forced nearly to the top of the funnel tube; then quickly close the rubber tube either by tightly pinching it or by pressing the tongue firmly against its end. If the apparatus is air-tight, the water in the funnel tube will not fall.

Now pour a few drops of the dilute sulfuric acid through the funnel tube *A* into the bottle *D*. Hydrogen is at once evolved. Enough of the acid must be added from time to time to cause a gentle and continuous evolution of the gas. An excess of the acid should be avoided, however, or the action will become too violent and a large quantity of zinc will have to be added at the close of the exercise. Pure sulfuric acid does not act readily on pure zinc; hence the addition of the copper sulfate, which serves to start the reaction.

It is evident that the gas which passes over first is a mixture of hydrogen and air. *The student must remember that such a confined mixture of hydrogen and air explodes with great violence if ignited.* Hence see that the end of the delivery tube is not brought near any flame. Determine when the hydrogen is free from air by repeatedly collecting a test tube full of gas and igniting it, holding the tube mouth downward. If pure, the gas burns quietly; otherwise there is a slight explosion. After all the air has been expelled from the generator, collect four 250-cc. wide-mouthed bottles of the gas. (12) What is the source of the hydrogen? (13) What is the use of the zinc?

Remove the cork from the generator, add a few more pieces of zinc, and set aside for ***e***. Sufficient zinc should be used so that a small portion of it remains undissolved.

***d.*** *Properties of hydrogen.* Thrust a lighted splint into a bottle of the gas held mouth downward. Slowly withdraw the splint and again thrust it into the gas. (14) Describe the results. (15) What do they prove?

Fill a small (60-cc.) wide-mouthed bottle or test tube one third full of water and invert it in a pneumatic trough. Displace the remaining water with hydrogen from one of the bottles. (16) What does the bottle now contain? Withdraw it from the water and, holding it at arm's length, quickly bring it, mouth downward, over a flame. (17) What do the results prove?

Uncover (mouth upward) a bottle of the gas. After a minute, test for the presence of hydrogen with a lighted splint. Repeat, holding the bottle mouth downward. (18) What do the results prove?

***e.*** *Recovery of the dissolved product.* Without removing the fragments of undissolved zinc, pour the liquid set aside in experiment ***c*** into an evaporating dish and boil the liquid gently on a ring-stand support. As soon as white crusts begin to form on the side of the dish, just above the liquid, filter the hot liquid into a beaker and set it aside to cool. (19) How does the product which separates from the filtrate compare in properties with the original zinc? (20) What is the product (pages 52–53 of text)? (21) How does it differ in composition from sulfuric acid?

## EXERCISE 9

## THE COMBUSTION OF HYDROGEN

**Object.** To learn how to burn hydrogen with safety and to identify the product of its combustion.

**Apparatus.** Hydrogen generator and tubes as shown in Fig. 35 (*A* is the hydrogen generator attached by rubber tubing *E* to a drying tube *B* (this tube is filled with granulated calcium chloride held in place by loose plugs of cotton placed at each end of the tube); *C* is a glass tube drawn to a jet); oxyhydrogen blowpipe; blast lamp; beaker.

**Materials.** Granulated calcium chloride sufficient to fill tube *B* (Fig. 35); cotton; 8 g. of mossy zinc; copper sulfate solution (R.S.); dilute sulfuric acid for generating hydrogen (see Exercise 8); picture-frame wire 10 cm. long; bit of charcoal.

***a.*** Charge the generator *A* (Fig. 35) with 6 or 8 g. of zinc, add a few drops of a solution of copper sulfate, cover with water, and add dilute sulfuric acid as in *c*, Exercise 8. Insert a piece of rubber tubing over the tube *C*, collect samples of the gas in test tubes over water, and test with a flame to see whether the hydrogen is free from air. *After all the air has been expelled, wrap a towel carefully about the generator* and

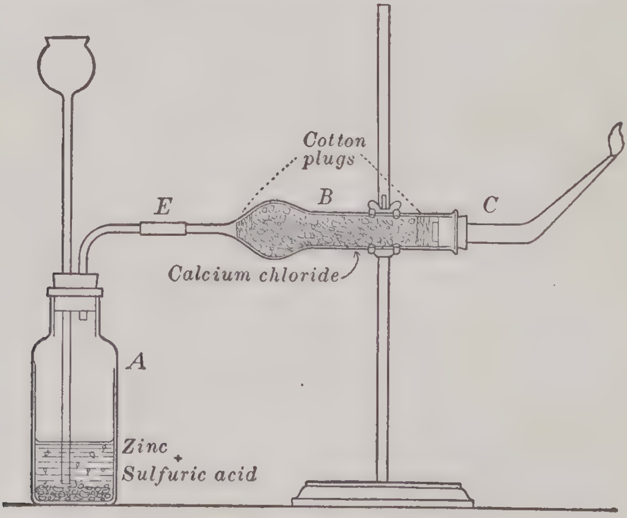

FIG. 35

cautiously ignite the hydrogen escaping from the jet *C*. The flame is nearly invisible and is very hot. Test the heat of the flame by holding in it different objects, such as a splint, a piece of picture-frame wire, a bit of charcoal.

Finally, hold over the flame a cold, dry beaker or bottle and note the liquid deposited on the sides of the vessel. (1) Explain. (2) Why is the gas passed through the tube *B*?

*b*. Examine the structure of the oxyhydrogen blowpipe. Compare it with the blast lamp used in the laboratory. (3) Draw a diagram representing a cross section of the blast lamp.

## EXERCISE 10

## OXIDATION AND REDUCTION

**Object.** To study the changes involved in the processes of oxidation and reduction.

**Apparatus.** Hydrogen generator and tubes as shown in Fig. 36 (*A* is the hydrogen generator, *B* is a drying tube filled with calcium chloride, *C* is a straight glass tube, and *D* is a hard-glass test tube); burner; apparatus used in preparing oxygen (Fig. 29).

**Materials.** 2 g. of copper oxide; enough calcium chloride to fill the drying tube *B*; 8 g. of mossy zinc; dilute sulfuric acid for preparing hydrogen (see Exercise 8); 4 g. of potassium chlorate; 2 g. of manganese dioxide.

*a*. Remove the tube *D* (Fig. 36), introduce into it 1 or 2 g. of copper oxide, and return it to the position shown in the figure.

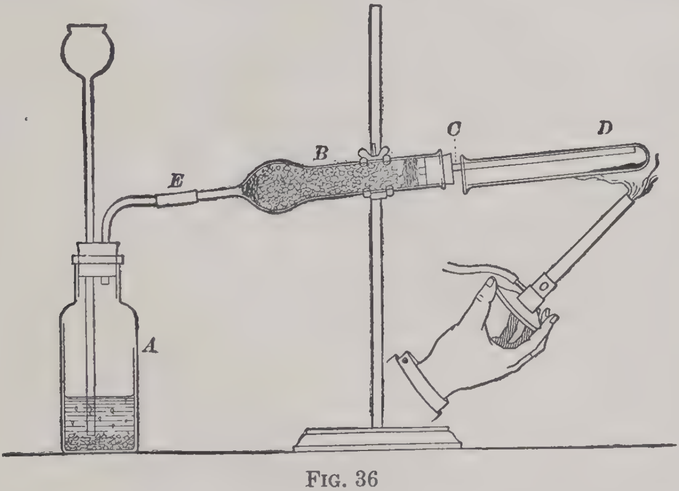

FIG. 36

Now generate hydrogen in *A*, as in Exercise 8. After all the air has been expelled from the apparatus and the generator wrapped in a towel (see Exercise 9), cautiously heat the copper oxide, being careful to keep the flame away from the mouth of the tube *D*. Note the condensation of moisture in the cold portions of the tube. (1) Is there any visible evidence of change in the copper oxide? (2) Explain. (3) What is the object of the calcium chloride in tube *B*?

*b.* Disconnect the bottle *A* at *E* from the rest of the apparatus and blow a little air through the tube *B* to remove the hydrogen present. Now connect the apparatus used in the preparation of oxygen (Fig. 29) at *E* to the tubes *B*, *C*, and *D*. Generate oxygen and conduct a slow current of the gas through *B*, *C*, and *D*, at the same time heating the residue in *D*. (4) Note and explain the changes that take place.

## EXERCISE 11

## THE MEASUREMENT OF GAS VOLUMES

**Object.** To learn how to measure gas volumes and to calculate the volumes which gases, measured under laboratory conditions, would occupy under standard conditions.

**Apparatus.** Graduated tube and cylinder arranged as shown in Fig. 37; thermometer; barometer.

*a.* (It is suggested that the instructor arrange one or more pieces of apparatus as shown in Fig. 37. The students will then take the readings and solve the problems.)

Partly fill a graduated tube *A* with water and invert it in a cylinder (or other vessel) of water as shown in Fig. 37. Let the water stand until its temperature is the same as that of the air. Adjust the tube until the level of the liquid inside and outside of the tube is the same; then take the reading of the volume of the air in the tube. Note the temperature of the air (place a thermometer by the side of the tube) and likewise the pressure of the atmosphere as indicated by the barometer. Record these values in their appropriate places in the following table; then calculate the volume which the air would occupy under standard conditions.

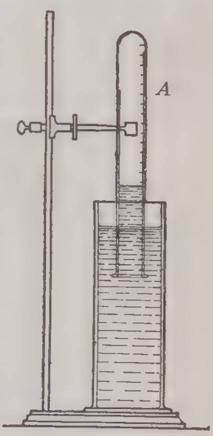

Fig. 37

1. Volume of air in tube . . . . . . . . . . . . . . . . cc.
2. Temperature of air . . . . . . . . . . . . . . . . . °
3. Barometric pressure . . . . . . . . . . . . . . . . mm.
4. Vapor pressure of water at temperature of air (see Appendix) mm.
5. Effective pressure on air in tube (barometric pressure less vapor pressure) . . . . . . . . . . . . . . . . mm.
6. Volume which the air would occupy under standard conditions (Calculate) . . . . . . . . . . . . . . . . cc.
7. Weight of above volume of air (see Appendix for weight of 1 liter under standard conditions) . . . . . . . . . g.

*b.* Suppose that the gas in the graduated tube (Fig. 37) were oxygen, (1) calculate its weight. Suppose it were hydrogen, (2) calculate its weight. (3) Calculate the weight of the water vapor in the tube *A*.

## EXERCISE 12

### SOME QUANTITATIVE RELATIONS OF OXYGEN

**Object.** To determine the weight of 1 liter of oxygen; also to determine the percentage of oxygen in potassium chlorate.

**Apparatus.** Apparatus shown in Fig. 38 (*A*, *B*, *C* is the apparatus used in the preparation of oxygen (Fig. 29); care must be taken that the glass tube and cork fit the test tube *air-tight*, so that any gas generated in the tube can escape only through the exit tube *C*; *D* is an ordinary 5-pint acid bottle, which must be completely filled with water and inverted in the water in the trough; the end of the exit tube *C* must extend so far up in the bottle *D* that it will be well above the level of the liquid when the gas is all evolved; in this way the gas in bottle *D* will be in continuous connection with the gas in tube *A* through the rubber tube *C*); cork to fit the bottle *D*; porcelain crucible; iron wire 10 or 12 cm. long, or file; graduated cylinder.

**Materials.** 3 g. of powdered manganese dioxide; 7 g. of potassium chlorate.

*a. Principle involved.* The molecular weight of a gaseous element or compound is determined by obtaining the weight of a definite volume of the gas. Since the direct weighing of a gas is not easily carried out, it is better to adopt some method which avoids the direct weighing of the gas. In the case of oxygen this is readily done by selecting a solid compound of oxygen (such as potassium chlorate) which will evolve the gas when heated. The difference in the weights of the compound before and after heating gives the weight of the oxygen evolved. By collecting the oxygen evolved it is easy to determine its volume. We have then the volume occupied by a definite weight of oxygen, and from these results we can calculate the weight of 1 liter of the gas. Proceed as follows:

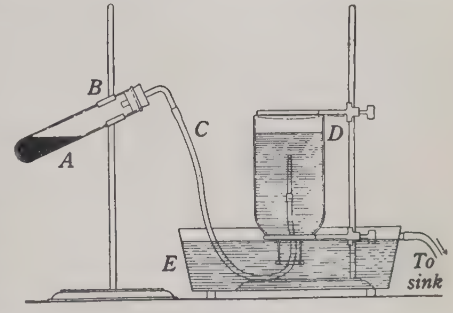

FIG. 38

*b. Preparation of the oxygen.* In your porcelain crucible place about 3 g. of finely powdered manganese dioxide and heat it strongly in the Bunsen flame for about five minutes, stirring it from time to time with a clean iron wire or the end of a file; then withdraw the flame and allow the powder to cool.

Thoroughly clean and dry your hard-glass test tube *A* (Fig. 38) and weigh it accurately to 0.01 g. Record the weight in the table at the end of this exercise. Weigh out roughly between 6 and 7 g. of potassium chlorate and introduce this into the tube (Fig. 26), taking care that none of the chlorate adheres to the sides of the tube. Weigh accurately (to 0.01 g.) the tube and contents as before, recording the weight in the table. Now introduce into the tube the manganese dioxide contained in the crucible, and again weigh to 0.01 g. and record the weight in the table. Mix the two compounds in the bottom of the tube by gently rotating the tube and by tapping it against your hand. Connect the tube as shown in Fig. 38 and arrange the exit tube so that all gas evolved will be collected in the bottle *D*, which must be completely filled with water. Now hold the burner in your hand and heat the mixture gently. Oxygen is evolved and is collected in the bottle. Regulate the flame so that the bubbles of the gas *can easily be counted*, say, about one each second. Continue the heating *until the gas is no longer evolved.* Then allow the whole apparatus to stand until it acquires room temperature.

Now withdraw the tube *C* from the bottle *D* and adjust the bottle so that the level of the water inside and outside the bottle is the same (it may be necessary to add more water to the trough); then, while holding it in this position, push a cork tightly into the bottle and quickly bring the bottle to an upright position on the desk. Next, take the temperature of the water in the trough *E* and the reading of the barometer and record the figures in the table. If time is not available, the bottle containing the oxygen (and some water), and the tube *A* in which the oxygen was prepared, can be set aside and the experiment concluded later.

*c. Determination of the weight of 1 liter of oxygen.* Weigh to 0.01 g. the test tube and residue left after preparing the oxygen, recording the weights in the table. The loss in the weight of the tube and contents through heating equals the weight of the oxygen evolved. Now measure the volume of the oxygen in the bottle by adding water from the graduated cylinder and record the volume. The volume occupied by the oxygen is under laboratory conditions. Reduce this to standard conditions as described on page 70 of text and record the result in the table. You now have the volume under standard conditions occupied by a known weight of oxygen. From these results calculate the weight of 1 liter of oxygen under standard conditions. Record the weights in the table.

*d. Calculation of percentage of oxygen in potassium chlorate.* If the directions are closely followed, all the oxygen evolved in the above experiment comes from the potassium chlorate; moreover, if the experiment is carried out as described, all the oxygen in the potassium chlorate will be evolved, since the mixture is to be heated until the gas ceases to be given off. From the weight of the potassium chlorate taken

and the weight of the oxygen evolved, calculate the percentage of oxygen in the potassium chlorate in accordance with the directions at the end of the table and record your results.

TABLE OF RESULTS

| | |
|---|---|
| 1. Weight of test tube *A* . . . . . . . . . . . . . . . . . | g. |
| 2. Weight of test tube plus potassium chlorate . . . . . . | g. |
| 3. Weight of potassium chlorate taken (equals the difference of the two weights given above) . . . . . . . . . . . . | g. |
| 4. Weight of the test tube plus potassium chlorate plus manganese dioxide . . . . . . . . . . . . . . . . . . . . | g. |
| 5. Weight of the test tube and residue left after heating . . | g. |
| 6. Weight of oxygen evolved (equals loss in weight of tube and contents by heating) . . . . . . . . . . . . . . . . | g. |
| 7. Temperature of water in pneumatic trough . . . . . . . | ° |
| 8. Reading of barometer . . . . . . . . . . . . . . . . . | mm. |
| 9. Volume of oxygen in bottle *D* . . . . . . . . . . . . | cc. |
| 10. Volume of this oxygen under standard conditions (Calculate) | cc. |
| 11. Weight of 1 liter of oxygen under standard conditions (Calculate) . . . . . . . . . . . . . . . . . . . . | g. |
| 12. Percentage of oxygen in potassium chlorate (Calculate from the following formula): | |
| $\frac{\text{Weight of oxygen evolved} \times 100}{\text{Weight of potassium chlorate taken}} = \left\{\begin{array}{l}\text{percentage of oxygen}\\ \text{in potassium chlorate}\end{array}\right\}$ | % |

| | ACTUAL | RESULTS OBTAINED | AVERAGE OF RESULTS OBTAINED BY CLASS |
|---|---|---|---|
| 13. Weight of 1 liter of oxygen . . . . | 1.4290 g. | | |
| 14. Percentage of oxygen in potassium chlorate . . . . . . . . . . . | 39.2 | | |

(1) What are the sources of error in the above experiment?

## EXERCISE 13

## DISTILLATION

**Object.** To find how to distill liquids and to learn the uses of distillation.

**Apparatus.** 2 flasks (250-cc.); condenser and connections as shown in Fig. 39; ring stands; wire gauze 12 cm. square; watch glass; funnel; evaporating dish.

**Materials.** 10 cc. of alcohol; distilled water; hydrant or well water.

*a.* (Two or more students may work together if the apparatus is not available for each.) Connect a Liebig condenser *B* with a 250-cc. flask *A*, as represented in Fig. 39. The flask is set on a piece of wire gauze supported by the iron ring attached to the ring stand. The rubber tube *C* is connected with the water pipe by means of rubber tubing, and a current of cold water is conducted through the outer tube of the condenser, escaping through the rubber tube *D*. (1) Why is cold water admitted at *C* rather than at *D*?

Fill the flask one fourth full of hydrant or well water and boil until 50 cc. or more of liquid has collected in the *receiver E*. The process of boiling a liquid and condensing its vapor is called *distillation.* (2) Compare the *distillate* (distilled water) with the hydrant water in appearance and in taste.

Place 4 or 5 drops of the distilled water on a watch glass and evaporate, holding the watch glass 10 or 15 cm. above the tip of the flame. (3) Is there any residue? (4) Repeat, using hydrant water. (5) Why is distilled water used in the laboratory?

*b.* Repeat the distillation, using a sample of muddy water in *A*. (6) Describe your results.

*c.* Pour 1 or 2 cc. of alcohol into an evaporating dish and bring a flame in contact with it. (7) Is the alcohol inflammable? Now distill a mixture of 10 cc. of alcohol (boiling point 78.5°) and 30 cc. of water. Collect the first 1 or 2 cc. of the distillate in an evaporating dish and test with a flame. In the same way test successive portions of the distillate. (8) Does there seem to be a partial separation of the two liquids? In this way a mixture of liquids boiling at different temperatures may often be separated more or less perfectly. The process is termed *fractional distillation.*

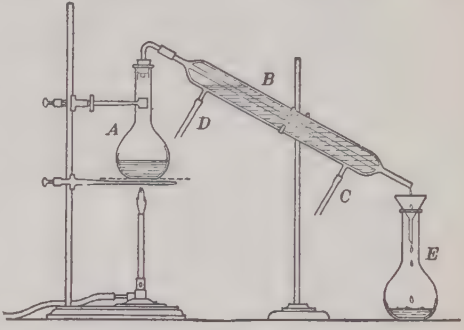

FIG. 39

## EXERCISE 14

## THE COMPOSITION OF WATER

**Object.** To determine the percentage composition of water; that is, the percentages of oxygen and hydrogen which it contains.

**Apparatus.** Hydrogen generator and tubes as shown in Fig. 40 and described below; balance.

**Materials.** 10 g. of mossy zinc; 5 g. of black powdered copper oxide; dilute sulfuric acid for generating hydrogen (Exercise 8); granular calcium chloride enough to fill tubes *B* and *C*.

*a.* Recall the experiments included under Exercises 9 and 10. (1) What do the results show with reference to the composition of water? (2) Is the process employed in Exercise 10 one of synthesis or of analysis? (3) Are the results qualitative or quantitative in character?

*b.* The experiments included under Exercise 10 may be modified in the following way so as to give quantitative results. Arrange an apparatus as shown in Fig. 40, in which *A* represents the hydrogen generator and *B* and *C* tubes filled with dry granular calcium chloride. *D* is the hard-glass test tube used in preparing oxygen (*A*, Fig. 29). This tube is fitted with a two-holed rubber stopper fitted with glass

tubes as shown in the figure. The U-tube *C* is fitted with one-holed stoppers, through each of which passes a short glass tube, the outer ends of which are fitted with short rubber tubes *F* and *G*.

Remove the hard-glass test tube *D* and introduce into it about 4 or 5 g. of black powdered copper oxide. Weigh the tube and copper oxide accurately to 0.01 g. and record the weight in the table on page 32. Now hold the tube in a nearly horizontal position and rotate it so that the copper oxide forms a little layer along the side of the lower half of the tube as shown in the figure; then connect the tube as shown in the figure, being careful that no copper oxide comes in contact with the long glass tube extending into *D*. Next disconnect the tube *C* at *F*, leaving the rubber tube *F* connected with *C*. In order to prevent the moisture of the air from being absorbed by the calcium chloride in *C*, the tubes *F* and *G* are closed by inserting into each a short glass tube closed at one end. The tube *C*, so closed, is then weighed to 0.01 g. and the weights recorded in the table.

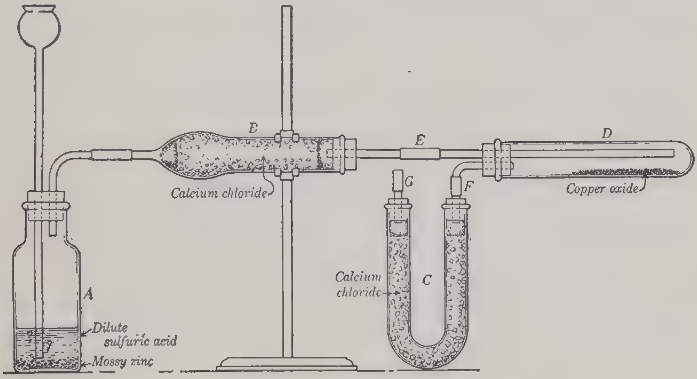

FIG. 40

Now remove the sealed tubes from the rubber tubes *F* and *G*, laying them aside so that they may be inserted again when the tube is reweighed; then connect the apparatus as shown in Fig. 40, taking care to render it air-tight. Generate hydrogen slowly, and *when the apparatus is free from air* ((4) How prove this?), heat the copper oxide in tube *D* very gently, using the wing-top burner. Gradually increase the heat (but not to redness), all the time maintaining the slow current of hydrogen. When the copper oxide is reduced, or nearly so (this should not take more than ten or fifteen minutes of gentle heating), withdraw the heat *but maintain the current of hydrogen until the apparatus is at room temperature.* If any of the water formed remains condensed in the end of tube *D*, a very gentle heat is cautiously applied until it is driven into the tube *C*.

When the apparatus has acquired the room temperature, pour out the contents of the bottle *A* and connect the empty bottle again as shown in the figure. Now pour a slow stream of water through the funnel tube into the bottle *A*. This forces a slow current of air through the entire apparatus, so that the hydrogen present is displaced by air, with which the apparatus was filled at the beginning of the experiment. When the bottle *A* is nearly filled with water, empty it; then connect again and fill it a second time with water. Enough air is thus forced through the apparatus to displace the hydrogen. Now disconnect the tubes *D* and *C*; also insert the sealed tubes in the rubber tubes *F* and *G*. Then weigh the tube *D* and contents to 0.01 g.; also the tube *C*, as in the beginning of the experiment, to 0.01 g., recording all weights in the table. Next make the calculations called for in the table:

Table of Results

| | |
|---|---|
| 1. Weight of tube *D* and contents before heating . . . . . . | 71.6 g. |
| 2. Weight of tube *D* and contents after heating . . . . . . . | g. |
| 3. Weight of oxygen in water formed (difference between weights 1 and 2) . . . . . . . . . . . . . . . . . | g. |
| 4. Original weight of tube *C*, contents, stoppers, and sealed tubes . . . . . . . . . . . . . . . . . . . . . . . | g. |
| 5. Final weight of tube *C*, contents, stoppers, and sealed tubes . . . . . . . . . . . . . . . . . . . . . . . | g. |
| 6. Water formed (difference between weights 4 and 5) . . . . | g. |
| 7. Percentage of oxygen in the water (equals weight of oxygen as recorded in 3 divided by weight of water as recorded in 6, multiplied by 100) . . . . . . . . . . . . . . . | % |
| 8. Percentage of hydrogen in the water (equals 100% – percentage of oxygen) . . . . . . . . . . . . . . . . . | % |

| | Actual | Results Obtained | Average of Results Obtained by Class |
|---|---|---|---|
| 9. Percentage of oxygen . . . . . . . . . | 88.81 | | |
| 10. Percentage of hydrogen . . . . . . . . | 11.19 | | |

(5) Discuss the sources of error in the experiment.

## EXERCISE 15

### HYDROGEN PEROXIDE

**Object.** To prepare hydrogen peroxide and to study its properties.

**Apparatus.** Test tube; 200-cc. beaker; glass rod; funnel and filter paper.

**Materials.** 10 cc. of ordinary hydrogen peroxide solution; wooden splint; 1 g. of manganese dioxide (powder); starch paste (R.S.); crystal potassium iodide; 6 cc. of ether; potassium dichromate (R.S.); 1 g. of sodium peroxide; ice water (100 cc.); piece of blue litmus paper; about 1 cc. of sulfuric acid, slowly added (*Caution*) to 5 cc. of water.

*a*. (1) What is the strength of the hydrogen peroxide solutions sold by the druggist (page 92 of text)? Pour 3 cc. of the solution into a large test tube and add 1 g. of finely powdered manganese dioxide. With a glowing splint test the gas evolved. (2) What is it? (3) What kind of agent is hydrogen peroxide?

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O. A.

Filter the mixture remaining in the tube. The solid is the unchanged manganese dioxide. (4) In what other experiment has manganese dioxide been used to assist in bringing about a chemical action, the dioxide, apparently at least, undergoing no change?

*b.* To 1 cc. of starch paste add 4 cc. of water. Dissolve in this a small crystal of potassium iodide and then add a few drops of a solution of hydrogen peroxide. The peroxide liberates the iodine from the potassium iodide and the resulting free iodine colors the starch paste. (5) Note the results.

*c.* Pour 3 cc. of the solution of hydrogen peroxide into a test tube and add an equal volume of ether. (*Caution. Ether is very inflammable. Keep it away from all flames.*) Shake the solution vigorously and notice that the ether quickly rises to the top of the tube when the tube is set aside. Now add 1 drop of a solution of potassium dichromate and again shake the mixture. (6) Now note the color of the layer of ether. A blue color in the ether constitutes a delicate test for hydrogen peroxide.

*d.* When sodium peroxide is added to water, hydrogen peroxide is formed, but this decomposes even at ordinary temperatures into water and oxygen (page 34 of text). If the temperature is kept low, however, the hydrogen peroxide remains unchanged, and it is possible in this way to prepare the compound. To prepare the peroxide proceed as follows: Add 1 g. of sodium peroxide, a little at a time, to 100 cc. of ice water. Now add dilute sulfuric acid, a few drops at a time, to the liquid (stir thoroughly with a glass rod) until a drop of the resulting liquid placed on a piece of blue litmus paper changes the blue to a red color. Then test the liquid for the presence of hydrogen peroxide in accordance with the methods given above under *b* and *c*. (7) Describe the results obtained.

## EXERCISE 16

## COMBINING WEIGHTS

**Object.** To determine the combining weights of some typical elements.

**Apparatus.** Porcelain crucible and cover; pipestem triangle; ring stand and burner; forceps.

**Materials.** From 0.5 to 1 g. of silver oxide; 0.1 g. of powdered silver (see Appendix for preparation of silver oxide and powdered silver); 20 cc. of concentrated chlorine water; 3 g. of granular lead; 2 g. of powdered sulfur.

NOTE. In this exercise it is desirable to divide the class, one experiment being assigned to each group.

*a. The ratio in which hydrogen combines with oxygen.* (1) What is meant by the term *combining weight*? (2) Referring to the results obtained in Exercise 14, calculate the number of grams of hydrogen that are combined with 8 g. of oxygen in water. (3) Does this calculation imply a knowledge of atomic weights or is it a preliminary step in the determination of atomic weights?

*b. The ratio in which silver combines with oxygen.* Silver oxide is easily decomposed into silver and oxygen on heating. By weighing a definite quantity of silver oxide, decomposing this by heat, and then weighing the resulting silver, it is easily possible to determine the ratio in which the silver and the oxygen are combined in silver oxide. Proceed as follows: Weigh a porcelain crucible and cover accurately to at least 0.01 g. and record the weight in the table below. Introduce into the crucible about 1 g. of silver oxide and again accurately weigh, recording the weight in the table. Place the crucible and cover on a pipestem triangle and apply a very gentle heat, being careful not to allow the tip of the flame to reach the crucible. After four or five minutes gradually increase the heat until the lower part of the crucible is heated nearly to redness. Continue the heating until the silver oxide is completely decomposed, the black color of the oxide giving way to the gray crystalline silver. Withdraw the heat and when the crucible has reached room temperature reweigh, again recording the weight. From your results calculate the number of grams of silver that are combined with 8 g. of oxygen in the silver oxide.

TABLE OF RESULTS

| | |
|---|---|
| 1. Weight of crucible and lid . . . . . . . . . . . . . . . . | g. |
| 2. Weight of crucible, lid, and silver oxide . . . . . . . . . | g. |
| 3. Weight of silver oxide (Calculate) . . . . . . . . . . . . . | g. |
| 4. Weight of crucible, lid, and silver . . . . . . . . . . . . | g. |
| 5. Weight of silver in the silver oxide heated (Calculate) . . . | g. |
| 6. Weight of oxygen in the silver oxide heated (Calculate) . . | g. |
| 7. Number of grams of silver combined with 8 g. of oxygen in silver oxide (Calculate from above results) . . . . . . . | g. |

*c. The ratio in which silver combines with chlorine.* The gaseous element chlorine combines readily with silver to form silver chloride. By letting chlorine act upon a definite weight of silver and accurately weighing the resulting silver chloride, the ratio in which silver and chlorine combine may be calculated at once. Proceed as follows: Weigh a porcelain crucible and cover accurately to at least two decimal places, recording the weight in the table on page 35. Place not less than 0.1 g. of powdered silver in the crucible, weigh again, and record the weight. To the crucible add 15 cc. of strong chlorine water, cover the crucible with the lid, and set it aside in your locker under a large beaker (for protection) until the next period. Arrange a ring stand, pipestem triangle, and burner *in the hood,* place the crucible on the triangle (Fig. 30, but with the crucible in a vertical position), and slowly evaporate the contents of the crucible nearly to dryness, holding the burner in your hand and heating just short of boiling. When nearly

dry, add 5 cc. of chlorine water and again evaporate to complete dryness. Very carefully heat the crucible and lid until all moisture has been driven off, being careful not to heat the contents of the crucible much above 100°. Allow the crucible and lid to cool until they have reached room temperature, weigh, and record your weighing. From your results calculate the number of grams of silver that have combined with 1 g. of chlorine. If your results fall much below the ratio 1 g. of chlorine to 3 g. of silver, add more chlorine water to the crucible and again evaporate and weigh.

### Table of Results

1. Weight of crucible and lid . . . . . . . . . . . . . . . . g.
2. Weight of crucible, lid, and silver . . . . . . . . . . . . . g.
3. Weight of silver (Calculate) . . . . . . . . . . . . . . . g.
4. Weight of crucible, lid, and silver chloride . . . . . . . . . g.
5. Weight of silver chloride from silver used (Calculate) . . . g.
6. Weight of chlorine in silver chloride formed (Calculate) . . g.
7. Number of grams of silver combined with 1 g. of chlorine in silver chloride (Calculate) . . . . . . . . . . . . . . . g.

*d. The ratio in which lead combines with sulfur.* Lead and sulfur, when heated, readily combine to form lead sulfide. By starting with a definite weight of lead and weighing the resulting lead sulfide one can calculate directly the ratio in which the lead and sulfur combine. Proceed as follows: Weigh a porcelain crucible and cover accurately to at least two decimal places, recording the weight in the table below. Place about 3 g. of granular lead in the crucible, reweigh, and record the weight. Now cover the lead in the crucible with from 1 to 2 g. of powdered sulfur. Place the crucible on a pipestem triangle, cover it with the lid, and heat it very gently. Some of the sulfur combines with the lead, while the remaining portion is vaporized, escaping under the lid and burning with a characteristic sulfur flame. When the vapor begins to burn, withdraw the flame temporarily. Finally, continue the heating until all free sulfur has disappeared from the crucible, and cover. Care must be taken to keep the crucible covered during the heating, in order to prevent oxidation. After all free sulfur has disappeared, withdraw the flame and allow the crucible to reach room temperature, then reweigh and record the weight.

### Table of Results

1. Weight of crucible and lid . . . . . . . . . . . . . . . . g.
2. Weight of crucible, lid, and lead . . . . . . . . . . . . . g.
3. Weight of lead taken (Calculate) . . . . . . . . . . . . . g.
4. Weight of crucible, lid, and lead sulfide . . . . . . . . . g.
5. Weight of lead sulfide formed from the lead taken (Calculate) g.
6. Weight of sulfur in the lead sulfide formed (Calculate from above results) . . . . . . . . . . . . . . . . . . g.
7. Number of grams of lead combined with 1 g. of sulfur in lead sulfide (Calculate) . . . . . . . . . . . . . . . . . g.

## EXERCISE 17

### DETERMINATION OF MOLECULAR WEIGHTS

**Object.** To determine the molecular weight of a typical element and of a typical compound.

**Apparatus.** A thermometer with a range at least 10° below the freezing point of water; 2 beakers of about 250-cc. capacity; a funnel 10 or 12 cm. in diameter at the top; glass rod; towel; 100-cc. graduated cylinder.

**Materials.** Beaker of cracked ice, lumps the size of acorns; 150 g. of sucrose (familiarly known as cane sugar or beet sugar).

### I. METHOD BASED ON AVOGADRO'S PRINCIPLE

Recall the result obtained for the weight of 1 liter of oxygen (Exercise 12). From this calculate the molecular weight of oxygen in accordance with Avogadro's principle. (1) Compare the results so obtained with those given in the text. (2) From your result calculate the number of atoms in the oxygen molecule.

### II. METHOD BASED ON THE LOWERING OF THE FREEZING POINT

*a. Calibration of thermometer for the freezing point.* First calibrate your thermometer for the freezing point as follows: Fill a 250-cc. beaker with lumps of ice about the size of acorns. Wash the ice clean by covering it with distilled water, stirring with a glass rod, and pouring off the water. Wrap a towel around the beaker, then just cover the clean ice with distilled water and insert the thermometer until the zero mark is just below the surface of the mixture. Stir the mixture slowly with the thermometer. When the thermometer indicates a constant temperature note exactly the reading, being careful to estimate to tenths of a degree. (A small magnifying glass will be of assistance.) When reading the thermometer keep the eye on the same level as the meniscus of the mercury. The reading thus made will be the freezing point of water on your thermometer. Record this value in the table on page 37.

*b. The lowering of the freezing point of water by sucrose.* Place the funnel on a filter stand and set a dry 200-cc. beaker under it. The funnel stem should just touch the inside wall of the beaker. Weigh about 30 g. of sucrose with an accuracy of 0.01 g. Place the sucrose in a 250-cc. beaker and add 50 cc. of water. Stir until the sucrose is dissolved. Now add pieces of the washed ice about the size of acorns until the beaker is nearly filled. Stir this mixture slowly and carefully with your calibrated thermometer. At first the temperature will fall ((3) Why?); then it will rise slowly ((4) Why?). Read the lowest point as accurately as possible to tenths of a degree and insert the value in the

table below. This reading indicates the freezing point of the solution. Immediately pour the contents of the beaker into the funnel. After a few seconds measure accurately with a graduated cylinder the volume of the solution that has run through the funnel. Record this volume in the table below.

*c. Correction for increase in volume of solvent due to sucrose.* We now know the volume of the solution and the weight of sucrose in the solution. To determine the molecular weight of sucrose we must know the volume (or weight) of the water present in the solution. This is not the same as the volume of the solution, for the volume of water is increased when sucrose is dissolved in it. The volume of water present in the solution may be determined with sufficient accuracy as follows: Pour into a graduated cylinder a quantity of water approximately equal to the volume of the solution obtained in *b*, and carefully note the reading on the graduation. Add 50 g. of sucrose, completely dissolve it, and again note the reading. The difference between the two readings gives the increase in volume due to the sucrose. Insert this value in the table below. By subtracting this value from the volume of the solution obtained in *b*, the volume of the water in the solution is found. Record this value in the table.

TABLE OF RESULTS

| | (1) | (2) |
|---|---|---|
| 1. Freezing point of water . . . . . . . . . . . | ° | ° |
| 2. Freezing point of sucrose solution . . . . . . . | ° | ° |
| 3. Lowering of freezing point . . . . . . . . . . | ° | ° |
| 4. Weight of sucrose . . . . . . . . . . . . . | g. | g. |
| 5. Volume of solution . . . . . . . . . . . . . | cc. | cc. |
| 6. Increase in volume due to sucrose . . . . . . | cc. | cc. |
| 7. Volume of water present in the solution (Calculate) | cc. | cc. |

*d. Calculations.* (5) From the above data calculate the number of grams of sucrose present in 1000 cc. of the solvent. (6) Calculate the number of grams of sucrose that are necessary to lower the freezing point of 1000 g. of water to – 1.875° (see text, page 113). This number will be the same as the number representing the molecular weight of sucrose. The molecular weight of sucrose as determined by the most accurate methods is 342.17. (7) Compare your result with this number.

## EXERCISE 18

### DETERMINATION OF SIMPLEST FORMULAS

**Object.** To determine the formula of copper oxide.

**Apparatus.** The apparatus shown in Fig. 41 (this is the same as the apparatus shown in Fig. 40 except that the U-tube *C* is not used).

**Materials.** 4 g. of *finely powdered* black copper oxide; zinc and sulfuric acid for preparing hydrogen as in Exercise 8; granular calcium chloride enough to fill tube *B* (Fig. 41).

*Simplest formula for copper oxide.* Disconnect the tube *D* (Fig. 41); weigh it accurately to 0.01 g., recording the weight in the table (page 39). Next introduce into this tube about 4 g. of the *finely powdered* black copper oxide, reweigh the tube and contents, recording the weight in the table. Hold the tube in a nearly horizontal position and rotate it until the copper oxide forms a thin layer along the side of the lower half of the tube as shown in the figure. Then, while holding it in this position, connect it as shown in the figure, taking care that the copper oxide does not come in contact with the long glass tube extending into the tube *D*. Generate hydrogen slowly in *A*, and when the apparatus is *free from air*, wrap a towel about the bottle and heat the copper oxide in tube *D* very gently, using the wing-top burner. Gradually increase the heat somewhat (do not heat to redness), all the time maintaining the slow current of hydrogen. Continue until the copper oxide is all reduced and the resulting water entirely expelled from the tube *D*. This should not take more than ten or fifteen minutes of gentle heating. Now withdraw the heat but *maintain the current of hydrogen until the apparatus has reached room temperature.*

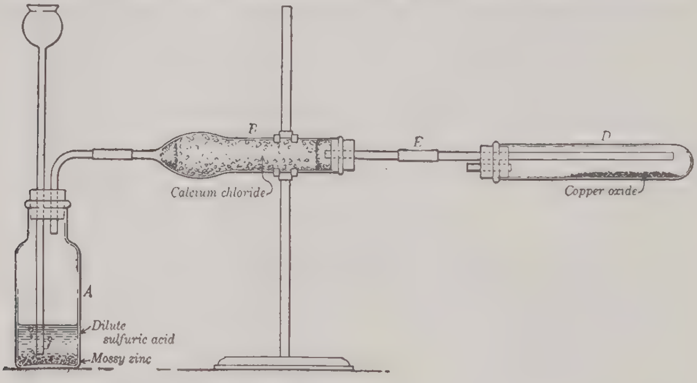


FIG. 41

When the apparatus has cooled to room temperature, disconnect the tube *D* carefully and hold it in a vertical position, mouth upward, for a

few seconds, so that the hydrogen in the tube will be displaced by air ((1) Why?), with which the tube was filled when originally weighed. Now again weigh the tube *D* and its contents accurately to 0.01 g., recording the weight in the table. From your results calculate (2) the percentage composition of copper oxide, and (3) also its simplest formula as directed in the following table:

Table of Results

| | |
|---|---|
| 1. Weight of tube *D* . . . . . . . . . . . . . . . . . . . . | g. |
| 2. Weight of tube *D* plus copper oxide . . . . . . . . . . . | g. |
| 3. Weight of copper oxide taken (difference between weights 1 and 2) . . . . . . . . . . . . . . . . . . . . . . . . . | g. |
| 4. Weight of tube *D* plus residue (copper) left after heating . . | g. |
| 5. Weight of copper left in tube (difference between weights 1 and 4) . . . . . . . . . . . . . . . . . . . . . . . . . | g. |
| 6. Percentage of copper in copper oxide as calculated from the following formula:<br>$\frac{\text{Weight of copper left in tube}}{\text{Weight of copper oxide taken}} \times 100$ . . . . . . . . . . . . . | % |
| 7. Percentage of oxygen in copper oxide (100% − percentage of copper in copper oxide) . . . . . . . . . . . . . . . | % |
| 8. Simplest formula (Calculate from percentage composition) . | |

(4) What additional data are necessary for the calculation of the exact (molecular) formula?

## EXERCISE 19

## THE WEIGHT OF A DEFINITE VOLUME OF STEAM; THE MOLECULAR FORMULA OF STEAM

**Object.** To determine the weight of a given volume of steam and to calculate from the result and that obtained in Exercise 14 the molecular formula of steam.

**Apparatus.** The apparatus shown in Fig. 42 and described on page 40. (Two students may work together. Since it is necessary to follow the directions carefully in order to obtain good results, it is essential that students carefully read the instructions, assemble the apparatus, and practice the manipulation involved before actually performing the experiment.)

*a. The principle involved.* A small volume of water is poured into a flask of known capacity. Heat is applied until the water is all boiled away. The resulting steam expels all the air from the flask, so that nothing remains in the flask but steam. The steam is then swept out of the flask by a current of dry air and absorbed by passing it through a tube filled with calcium chloride. The increase in the weight of this tube gives the weight of the steam. The temperature of the steam and the barometric readings must be taken so as to make the proper corrections for the volume of the steam. We now have the weight of a given volume of steam and from this can calculate the weight of 22.4 liters under standard conditions if it were to remain a gas.

*b. The apparatus.* The apparatus required is shown in Fig. 42: *A* is a 500-cc. pyrex flask, resting on a sand bath. The flask is fitted with a three-holed rubber stopper, through which is inserted the thermometer *B* and the two glass tubes *C* and *D*. The end of the tube *D* is bent and drawn out into a closed capillary. A scratch is made on the under part of this tube near the capillary end as shown in Fig. 42 (*a*), so that the end of the capillary may be broken easily at the proper time by pushing the tube *D* down against the side of the flask. In order to prevent any condensed steam (water) from collecting in the tube *C*, that portion of the tube outside the flask is bent so as to form an acute angle, and the end of the tube inside the flask is made somewhat jagged. Surrounding the flask and sand bath is a tin can *E* (about 2 liters in capacity) open at both ends; the flask, sand bath, and can are held in place by suitable clamps and ring stand, such as shown in Fig. 42; *F*, *G*, and *H* are drying tubes attached by cord or thin wire to the rod *I*, which is held in place by the clamp *J* (these tubes are filled with the materials indicated in the figure; care must be taken to use good materials, to see that the tubes are properly filled, and fitted with good stoppers, so that there will be no air leaks); *K* is a 250-cc. wide-mouthed bottle about one third full of sulfuric acid, and with connections as shown; *L* is a 5-pint acid bottle, fitted with a siphon tube, the rubber part of which is provided with a screw clamp *M*. Two pieces of asbestos paper are cut of such size and shape (*N*, *N′*, Fig. 42 (*b*)) as to fit over the top of the tin can and about the stopper of the flask *A*, and are fitted into place as shown in the figure.

Pour about 10 cc. of water into the flask *A*, connect the parts of the apparatus as shown in the figure, and test the apparatus to see that it is air-tight (see Exercise 27, ***a***).

*c. The procedure.* Disconnect the tube *G*, leaving the pure gum rubber connections *O*, *O′* attached to the tube. Insert into the end of each of these a glass seal (a short glass tube, sealed at one or both ends), so as to render the tube air-tight. Carefully wipe the tube with a clean cloth; then weigh the tube and contents to 0.01 g. (Place the values of all weights and volumes in the table at the end of the exercise.) Set the tube aside.

Now heat the sand bath, gradually raising the temperature, regulating the flame so that the thermometer registers about 140°. The water in the flask boils away and the resulting steam sweeps out the air in the flask through the tube *C*. The process of boiling away the water and replacing the air in the flask with steam will ordinarily require about thirty minutes. Its progress may be noted from time to time by slightly withdrawing one of the asbestos coverings *N*, *N′*. Continue the heating, maintaining the temperature at about 140° until all the water has boiled away. *It is absolutely essential for the success of the*

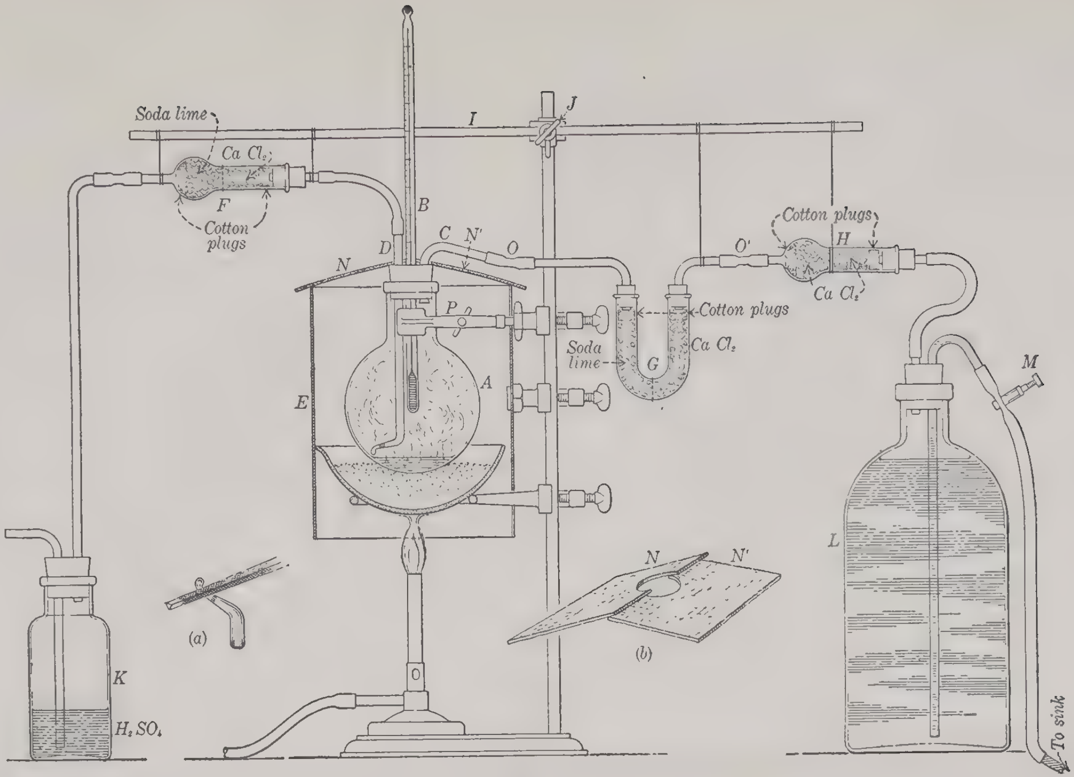

Soda lime
Ca Cl₂
F
Cotton plugs
I
J
B
D
N
C
N'
O
P
A
E
Soda lime
G
Cotton plugs
Ca Cl₂
O'
Cotton plugs
H
Ca Cl₂
M
L
N
N'
(a)
(b)
K
H₂ SO₄
To sink

FIG. 42

*experiment that no drops of water be left anywhere in the flask or in the exit tube C.* Care must be taken to have the surfaces of the clamp *P*, which are in contact with the flask, covered with asbestos paper; for any iron in contact with the neck of the flask serves to conduct away the heat, and the steam will condense at this point. Any drops of water that may remain in the outer part of the tube *C* may be expelled by a *small* Bunsen flame.

When the flask *A* and the tube *C* are *entirely free* from droplets of water and contain only *dry* steam (uniform heating must be continued throughout the experiment), proceed to determine the weight of this steam by absorbing it in the tube *G*. Preparatory to doing this slightly open the screw clamp *M* so that a *small* stream of water flows from the bottle *L*. Now remove the glass seals from the U-tube *G* (laying these aside for use when the tube is reweighed), and quickly connect it with the exit tube *C* and the drying tube *H*, as shown in the figure. Next break the capillary end of the tube *D* by carefully pressing the end against the side of the flask, using a gentle downward screw-like motion. *Note the exact temperature at this instant* and record it in the table. The heating is continued throughout the experiment. The water flowing from the bottle *L* will now draw through the apparatus a stream of air, dried by being bubbled through the sulfuric acid in *K*, and freed from carbon dioxide by the soda lime in *F*. The steam in the flask *A* is thus swept along and is absorbed as it passes over the contents of the U-tube *G*. Since the steam must all be absorbed, it is essential that the air be drawn through the tube at a gentle rate, say 2 or 3 bubbles per second (bottle *K*). This rate is controlled by the screw clamp *M*. At this rate, it will require about twenty minutes to sweep out all the steam from the flask. The U-tube *G* is then disconnected, the glass seals are inserted, and it is weighed to 0.01 g. This gain of weight is the weight of the steam originally contained in the flask *A*.

*d. The volume of the steam.* We now have the weight of the steam; it remains for us to determine the volume occupied by the steam. This equals the volume of the flask, with proper corrections for the space occupied by those parts of the stopper, the thermometer, and the tube *D*, which extended into the flask. Proceed as follows:

Place a strip of gummed label on the neck of the flask at the point that marks the depth to which the stopper is inserted. Now remove the stopper with accompanying tubes and thermometer. Measure the volume of the water necessary to fill the flask up to the point marked by the label and insert the value in the table. To determine the corrections to be made, fix the points on the tube *D* and the thermometer just below the stopper. This may be done by making a slight scratch on the tube *D* at this point, and, in the case of the thermometer, by noting the reading at this point. Now immerse each of these up to the points

designated in some water placed in a graduated cylinder (keep the end of your finger over the end of the tube *D* so that the water cannot enter the tube) and note the increase in the reading, recording the value. (1) From these data, calculate the volume occupied by the steam and insert the value in the table.

### TABLE OF RESULTS

| | |
|---|---|
| 1. Weight of tube *G* + contents + steam | g. |
| 2. Weight of tube *G* + contents | g. |
| 3. Weight of steam | g. |
| 4. Volume of flask *A* to the point marked by the strip of gummed label | cc. |
| 5. Corrections to be made for the space occupied by the thermometer and the tube *D* within the flask | cc. |
| 6. Volume of the steam under the conditions of the experiment | cc. |
| 7. Barometric pressure | mm. |
| 8. Temperature of steam in flask *A* at the instant when the capillary end of the tube *D* is broken | ° |
| 9. Volume of steam (standard conditions) (Calculate) | cc. |
| 10. The weight of 1000 cc. of steam under standard conditions (Calculate) | g. |
| 11. The molecular weight of steam | |

*e. Problem.* (2) From the molecular weight of steam and the data obtained in Exercise 14 calculate the exact (molecular) formula for steam. (3) Is the formula for steam necessarily the same as that for water?

## EXERCISE 20

## THE LAW OF MULTIPLE PROPORTION

**Object.** To secure proof of the law of multiple proportion.

**Apparatus.** The apparatus represented in Fig. 38, Exercise 12; the apparatus represented in Fig. 41, Exercise 18.

**Materials.** 6 g. of cuprous oxide (red oxide of copper); 6 g. of potassium perchlorate.

NOTE. In this exercise the class should be divided into two sections, with alternate assignments.

In preceding exercises it has been found possible to determine the exact weight of oxygen that can be obtained from a given weight of potassium chlorate; and to find the exact composition of a number of compounds (within the limits of experimental accuracy) and assign formulas to them. All these experiments afford proofs of the *law of definite composition*. In the present exercise similar proofs will be sought for the *law of multiple proportion*.

*a. Comparison of the weights of oxygen in the two oxides of copper.* In Exercise 18 the percentage of oxygen in the black oxide of copper was determined. Repeat this experiment in every detail, substituting about 6 g. of the *red oxide* of copper instead of the black oxide. From the two results, calculate the weight of oxygen combined with 10 g. of copper in each of the two compounds. (1) How do the two weights of oxygen compare with each other? (2) Do these results conform to the law of multiple proportion?

*b. Comparison of the weights of oxygen in potassium chlorate and potassium perchlorate.* In Exercise 12, *b* and *d*, the weight of oxygen in a weighed portion of potassium chlorate was found by heating the chlorate in a glass tube until no more oxygen was evolved, and then weighing the residue. Repeat this experiment in every detail, substituting about 6 g. of potassium perchlorate in place of potassium chlorate. For the purposes of this experiment it is not necessary to collect and weigh the oxygen evolved as described in Exercise 12, *c*, so that bottle *D*, Fig. 38, will not be needed. The tube *C* should dip under water in the trough *E* to show how rapidly the oxygen is being evolved and when the evolution stops. Heat the tube *A* carefully so as to maintain a steady but not too rapid evolution of oxygen. When oxygen ceases to come off, and while still applying heat, remove the stopper from the tube *A*, then allow the tube to come to the temperature of the room and accurately weigh it.

Both potassium chlorate and potassium perchlorate are compounds of potassium, chlorine, and oxygen. When either of these compounds is heated sufficiently, all the oxygen in it is given off as gas, and a compound of potassium and chlorine called potassium chloride is left as solid residue. (3) From the results obtained in Exercise 12 for potassium chlorate and from those obtained in the present experiment with potassium perchlorate, calculate the weights of oxygen in these two compounds combined with 10 g. of potassium chloride. (4) How are these two weights of oxygen related to each other? (5) Are they in accord with the law of multiple proportion?

# EXERCISE 21

## VALENCE

**Object.** To determine the valence of certain metals.

**Apparatus.** Apparatus arranged as in Fig. 43 (*A* is a wide-mouthed bottle holding about 150 cc.; *D* is a glass tube drawn out somewhat at the lower end and connected to the end of the funnel *B* by a rubber tube which is closed by the clamp *C*; the bottle *E* is the ordinary 5-pint acid bottle; *G* is a pneumatic trough); stopper for bottle *E*; graduated cylinder.

**Materials.** 2 g. of pure magnesium ribbon; 20 cc. of hydrochloric acid diluted with 20 cc. of water; 1.5 g. of pure aluminum turnings; 5 g. of pure mossy zinc.

NOTE. The valence of any element may be determined by finding out by experiment how many gram-atomic weights of hydrogen (or other univalent element) 1 gram-atomic weight of the element will combine with or displace in a compound. For example, we have learned that many of the metals, such as magnesium, zinc, and aluminum, displace the hydrogen from acids. It is evident that 1 gram-atomic weight of any of these metals when dissolved in an acid will displace 1, 2, 3, etc. gram-atomic weights of hydrogen, according to whether the metal is univalent, bivalent, tervalent, etc. Hence if we determine by experiment the weight of the hydrogen evolved by 1 gram-atomic weight of the metal, and divide this weight of hydrogen by 1.0078 (the gram-atomic weight of hydrogen), the quotient will be equal to the valence of the metal. Since it is impossible to eliminate all experimental errors, the quotient will not be an exact whole number (except by chance); but if the experiment is performed with care, the quotient will be so near to a whole number that no doubt will exist as to the integer which indicates the valence.

The class should be divided into three sections with appropriate assignments.

*a. The valence of magnesium.* Arrange an apparatus according to Fig. 43 as directed above under "Apparatus." Obtain about 2 g. of pure magnesium in the form of a ribbon and weigh it accurately to 0.01 g., recording the weights in the table on page 46. Introduce this into the bottle *A* (Fig. 43), and cover it with 10 cc. of water. Nearly fill the funnel *B* with the dilute hydrochloric acid prepared as directed above under "Materials." Before inserting the stopper in the bottle *A* place a small beaker under the tube *D* and partly open the clamp *C* until the rubber tube and the glass tube *D* are entirely filled with the dilute acid; then place the stopper firmly in the bottle *A*, taking care that it fits air-tight. The bottle *E* must be

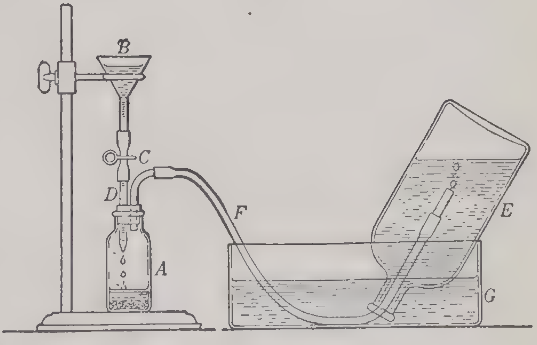


FIG. 43

completely filled with water, and the end of the exit tube *F* must extend at least two thirds of the way up into the bottle *E*, so that by the close of the experiment it will be well above the level of the water in the bottle *E*. In this way the gas collected in *E* will be in direct communication with the gas in the bottle *A*, through the rubber tube *F*. Now open the clamp *C* sufficiently to allow 4 or 5 drops of the dilute acid to enter the bottle *A*. Continue adding the acid, a few drops at a time, at such a rate as to produce a gentle evolution of gas. The funnel *B* must always contain a sufficient volume of the dilute acid to prevent any air bubbles from entering the bottle *A* with the acid. Continue the addition of acid from time to time until *all the metal* in the bottle *A* has been dissolved; then allow the entire apparatus to stand until it has reached room temperature. Withdraw the exit tube *F* from the bottle *E* and adjust the bottle *E* so that the level of the water in *E* is the same as the level of the water in the pneumatic trough *G*. While holding the bottle carefully in this position insert a stopper in the bottle *E*, then remove the bottle and stopper from the trough and place it in an upright position on the desk. Now measure the volume of the gas collected in the bottle *E* by adding water from a graduated cylinder; also measure the volume of the liquid that has collected in the bottle *A*, (1) inserting the values in the table below and making the calculations called for.

### TABLE OF RESULTS

| | |
|---|---|
| 1. Weight of metal taken | g. |
| 2. Volume of dilute acid added to *A* (this equals the total volume of liquid in *A*, less the volume of the water added before the apparatus was connected, namely 10 cc.) | cc. |
| 3. Volume of gas collected in *E* | cc. |
| 4. Volume of hydrogen evolved measured under laboratory conditions (this equals the volume of the gas collected in the bottle *E*, less the volume of dilute acid run into bottle *A*) | cc. |
| 5. Temperature of the gas in bottle *E* | ° |
| 6. Tension of aqueous vapor (see Appendix) | mm. |
| 7. Barometric pressure | mm. |
| 8. Volume that the hydrogen evolved would occupy under standard conditions (Calculate) | cc. |
| 9. Weight of this volume of hydrogen (Calculate from weight of 1 liter given in the Appendix) | g. |
| 10. Weight of hydrogen evolved by 1 gram-atomic weight, namely 24.32 g., of magnesium (Calculate) | g. |
| 11. Valence of the metal (Calculate by the following formula): | |

$$\text{Valence} = \frac{\text{weight of H evolved by 1 gram-atomic weight of metal}}{1.0078}$$

*b. The valence of aluminum.* Proceed exactly as in determining the valence of magnesium in experiment *a*, using about 1.5 g. of aluminum turnings in place of the magnesium. (2) Record your results.

*c. The valence of zinc.* Proceed exactly as in experiment *a*, using about 5 g. of mossy zinc in place of the magnesium. (3) Record your results.

## EXERCISE 22

## STATES OF MATTER

**Object.** To learn something of the properties of gases, liquids, and solids.

**Apparatus.** Watch glass; 250-cc. flask fitted with cork through which pass a thermometer and exit tube as shown in Fig. 44; thermometer graduated to at least 110°; ring stand and burner; 2 small beakers (or evaporating dishes); test tubes; glass rod; sand bath; small evaporating dish; beaker large enough so that, when inverted, it will fit over the evaporating dish.

**Materials.** 1 cc. of ether or chloroform; 10 cc. of benzene; pieces of ice; 100 g. of common salt; 5 g. of powdered sulfur; 3 g. of powdered alum; 1 g. of benzoic acid.

*a.* Place about 1 cc. of ether (***Caution.*** *Ether is inflammable*) or of chloroform on a watch glass and blow upon the surface of the liquid. (1) Account for the rapid evaporation. (2) Do you notice any change in the temperature of the watch glass? Explain.

*b.* Pour about 100 cc. of distilled water into the 250-cc. flask (Fig. 44). The lower part of the thermometer must dip into the liquid so that the temperature of the liquid may be noted. Now heat the water at such a rate that the temperature rises slowly but steadily. (3) At what temperature do you observe the formation of bubbles? (4) Where do they appear to form? (5) Of what are they composed? (6) At about what temperature do larger bubbles begin to form at the bottom of the vessel? (7) What becomes of them? (8) At what temperature do they move freely up through the liquid to the surface? (9) Do they get larger or smaller as they rise? Explain (page 139 of text). When the water is gently boiling, try increasing the heat. (10) Is the boiling any more energetic? (11) Does the temperature rise at all? (12) How do you define the boiling point?

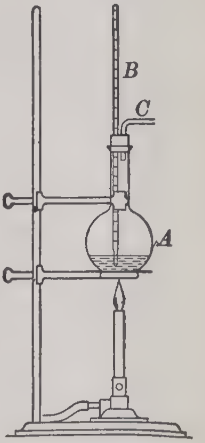


FIG. 44

*c.* Pour about 10 cc. of benzene into a clean, dry, hard-glass test tube. *This liquid is inflammable and must be kept away from all flames.* Pour into a beaker some ice water in which some pieces of ice are floating. In this ice water immerse that part of the test tube which is filled with benzene. Stir the benzene constantly with a thermometer, noting the temperature from time to time. (13) Carefully note the temperature at which the benzene begins to freeze, and continue the reading until the entire liquid, or nearly all of it, is frozen. Now remove the tube from the ice water and hold the lower part of it in the hand until the benzene is melted, noting the temperature during this change. (14) Note the results and discuss. (15) What becomes of the heat required to change a liquid such as water into a vapor?

*d.* Experiment *c* may be repeated, using water in the test tube and immersing it in a freezing mixture made by mixing 3 parts of crushed ice with 1 part of common salt.

*e.* Fill a test tube about one third full of powdered sulfur; then heat the tube gently until the sulfur is melted. Set the tube aside until the liquid has again solidified. (16) Is the solid in crystalline form?

*f.* Place about 3 g. of powdered alum in a small beaker or evaporating dish and dissolve the solid in as little water as possible, at room temperature. Hasten the solution by stirring the mixture with a glass rod. In the same way make a solution of common salt. Set the solutions aside (do not cover the beakers or dishes) until the next laboratory period. (17) Note the form in which each solid is deposited, illustrating, if possible, by a drawing. (18) Mention two common methods used for obtaining substances in a crystalline state.

*g.* Place a small evaporating dish on a sand bath. Weigh about 1 g. of benzoic acid (a white solid present in the resin which exudes from certain trees), and transfer it to the dish. Over the dish place an inverted beaker, the rim of which rests on the sand in the bath. Now heat the sand bath gently. The acid vaporizes, and the vapor, coming in contact with the cold side of the beaker, forms crystals. (19) What is this process called?

## EXERCISE 23

## PROPERTIES AND CONDUCT OF CARBON

**Object.** To prepare various forms of carbon and to study their properties.

**Apparatus.** Hard-glass test tube *A* connected as shown in Fig. 45; *B*, a test tube fitted with a glass tube *C* drawn to a jet; large beaker; burner; mortar and pestle; porcelain dish; funnel and filter paper; 250-cc. flask; glass rod.

**Materials.** Small pieces of hard wood (sawdust will do) sufficient to half fill the hard-glass test tube; pieces of soft coal sufficient to half fill the test tube; 3 g. of sugar; test tube one fourth full of boneblack; 1 cc. of litmus solution; 3 g. of copper oxide and an equal bulk of powdered charcoal; 1 cc. of limewater (R.S.).

*a.* Half fill the tube *A* (Fig. 45) with hard-wood splints or sawdust and connect it as shown in the figure. The tube *B* is kept cool by ice water in the beaker. Heat the wood in *A*, gradually increasing the heat until no further change takes place. (1) During the heating, test the gas escaping from the jet *C* to determine if it is combustible.

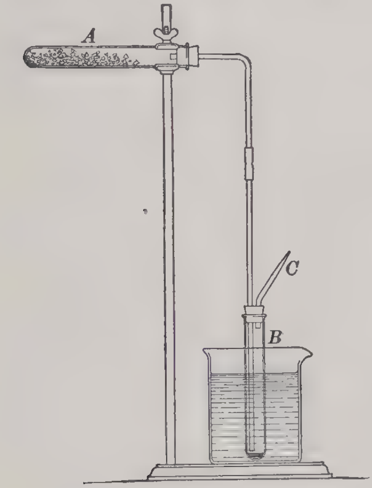


FIG. 45

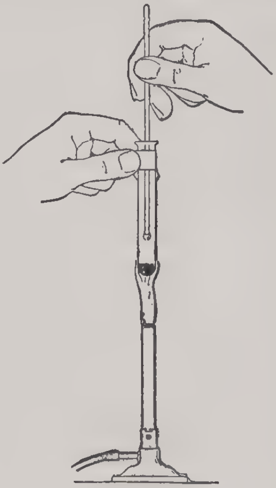


FIG. 46

When the tube *A* is cool, remove the residue. (2) What is it? (3) Note its properties. (4) Is it combustible? (5) Note the odor and appearance of the liquid condensed in *B*. (6) What name is applied to the process undergone by the wood (page 161 of text)?

*b.* Repeat experiment *a*, substituting small pieces of *soft* coal for the wood. (7) Describe the results. (8) What is left in the tube *A*? The liquid condensed in *B* contains *coal tar* (pages 468 and 492 of text). (9) Note its appearance and odor.

*c*. Heat 2 or 3 g. of sugar in a test tube until no further change takes place. (10) Note the results. (11) What is the residue?

*d*. Bring a cold porcelain dish into a small luminous Bunsen flame. Note the deposit. (12) What is this form of carbon called? (13) In what other forms does carbon exist?

*e*. Put into a small flask enough boneblack to fill a test tube one fourth full and pour over it about 50 cc. of water to which has been added a few drops of a solution of litmus or indigo. Thoroughly mix the contents of the flask; then heat gently for a few minutes and filter. If the filtrate is not decolorized, repeat the process, using more boneblack. (14) What is the composition of boneblack? (15) By what other name is it known? (16) What use does this experiment suggest for it?

*f*. Mix together in a mortar 2 or 3 g. of black copper oxide and an equal bulk of powdered charcoal. Transfer to a hard-glass test tube and heat gently. The hot carbon unites with the oxygen of the copper oxide, forming carbon dioxide. Test for the presence of carbon dioxide (*c*, Exercise 7) by holding in the tube (Fig. 46) a glass rod to the end of which is clinging a drop of clear limewater. (17) Complete and balance the following equation:

$$CuO + C \longrightarrow$$

(18) What kind of agent is hot carbon?

## EXERCISE 24

## CARBON DIOXIDE

**Object.** To prepare carbon dioxide and to study its properties.

**Apparatus.** Apparatus as shown in Fig. 47 (*A* is the *hard-glass* tube included in your outfit and is supported on a ring stand; *B* is a 1-liter (or larger) bottle (the ordinary 5-pint acid bottle serves the purpose well); the glass tube *D* extends to the bottom of the bottle *B* and is connected to a water tap by means of a rubber tube); 3 wide-mouthed bottles (250-cc.); hydrogen generator and connections as shown in Fig. 48; small beaker or test tube; hard-glass test tube.

**Materials.** 8 or 10 small pieces of charcoal (about the size of a bean); 5 pieces of marble (the size of small walnuts); hydrochloric acid; splints; limewater (R.S.).

*a*. *Preparation of carbon dioxide by direct combination of carbon and oxygen.* Arrange an apparatus as shown in Fig. 47. In the glass tube *A* place 5 or 6 small pieces of charcoal and connect the apparatus as shown. Pour about 10 cc. of clear limewater into the bottle *C*. Now turn the water cock slightly so that a slow current of water flows into the bottle *B*. This forces the air in the bottle out and over the charcoal in *A* and finally up through the limewater in *C*. Continue for one or two minutes. (1) Is there any change in the appearance of the lime-

water? Turn off the water and gradually heat the charcoal in tube A with the wing-top burner. When the charcoal is hot, continue the heating and again turn on the water so that a slow current of air is forced over the hot charcoal. (2) Note again any changes in the appearance of the limewater and account for them, writing the equations for the reaction (page 172 of text). (The amount of carbon dioxide in the air is so small that it has no appreciable effect on the limewater under the conditions of the experiment.)

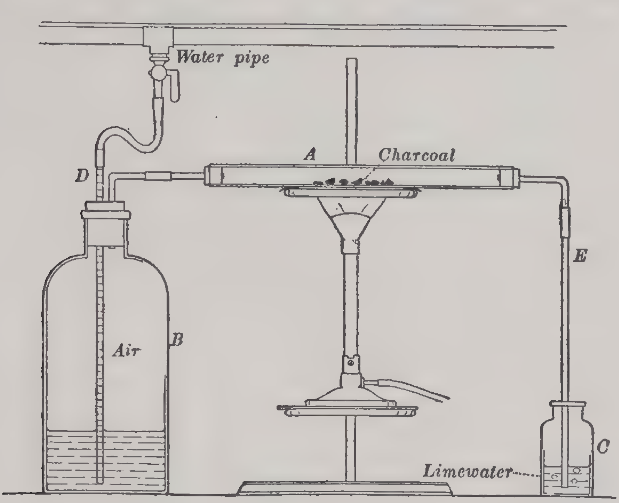


FIG. 47

*b. Preparation of carbon dioxide by the action of hydrochloric acid on marble (usual laboratory method).* In the laboratory, carbon dioxide is prepared by the action of an acid (hydrochloric serves best) on calcium carbonate, which has the formula $CaCO_3$. This occurs in many forms in nature, marble being the cheapest and most convenient form for the preparation of carbon dioxide. To prepare carbon dioxide by this method, proceed as follows:

Place some pieces of marble in your hydrogen generator and connect it as shown in Fig. 48. Add water through the funnel tube until the marble is covered with the liquid; then add hydrochloric acid, a few drops at a time. Collect three bottles of the gas by displacement of air. To test when filled, hold a burning splint at the mouth of the bottle; the gas will extinguish the flame. (3) Why not collect the gas over water, as in the case of oxygen and hydrogen? (4) Write the

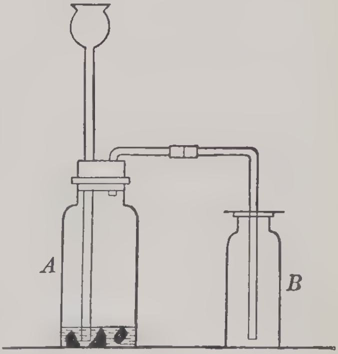


FIG. 48

equation for the reaction between the acid and the marble (page 169 of text). Calcium chloride ($CaCl_2$) is a white solid. (5) How could you recover the calcium chloride formed in the reaction?

*c. The properties of carbon dioxide.* (6) Note the color, odor, and taste of the gas. (7) Devise a simple experiment to prove whether the gas is heavier or lighter than air. Insert a burning splint in one of the bottles of the gas. (8) Is the gas combustible? (9) Is it a supporter of combustion? (10) The results suggest what use for the gas?

*d.* (11) What experiments have you performed that prove that carbon dioxide is present in the exhaled air? (12) that it is one of the products of combustion of ordinary fuels?

## EXERCISE 25

## CARBON MONOXIDE

**Object.** To prepare carbon monoxide and to study its properties.

**Apparatus.** The apparatus shown in Fig. 49 (*A* is a funnel, connected by a rubber tube with a piece of glass tubing which passes through a stopper into the 250-cc. flask *B*; *C* is a small pinch clamp); pneumatic trough; 3 wide-mouthed bottles; 3 glass plates 10 cm. square; apparatus shown in Fig. 50 (*A* and *B* are glass tubes connected by a rubber tube; *C* is a wide-mouthed bottle); wing-top burner.

**Materials.** Sulfuric acid; 25 cc. of formic acid (10-normal); limewater (R.S.); 1 g. of powdered black copper oxide; wooden splints.

***Caution.*** *Carbon monoxide is a nearly odorless and very poisonous gas and must not be breathed. All the experiments must be performed in the hood. After the gas is generated, pour the contents of the generator flask into a sink or a jar in the hood. If a good hood is not available, the exercise should be omitted.*

*a. Preparation of carbon monoxide.* Remove the stopper from the flask *B* (Fig. 49), pour in 15 cc. of sulfuric acid, and connect the apparatus as shown in the figure. Close the clamp *C* and partly fill the funnel *A* with the formic acid. Now open the clamp carefully so that the formic acid will enter the flask drop by drop. Allow 8 or 10 drops to flow in; then close the clamp. If the reaction does not begin (as indicated by absence of effervescence of the liquid in the flask and by the escape of gas through the exit tube), heat the flask very gently until the reaction starts; then open the clamp again and admit the formic acid, a drop at a time, so as to secure a regular flow of gas from the flask. If necessary, add more formic acid to the funnel, so as to keep it partly filled. Collect three bottles of the gas as shown in the figure, being careful to note which of the bottles was the first one filled. Close the clamp so as to stop the further generation of gas. Slip the glass plates over the mouths of the bottles and remove the bottles from the trough,

keeping them closed with the glass plates. (1) Write the equation for the preparation of the oxide from formic acid (page 165 of text).

*b. Properties and chemical conduct of carbon monoxide.* (2) Has the gas any color? (3) Is it soluble in water?

(4) Is the first bottle of gas collected pure carbon monoxide? Test it by bringing a burning splint to the mouth of the bottle; then repeat with the second bottle. (5) Is the gas combustible? (6) Is it a supporter of combustion? Slip the glass plate from the third bottle just far enough to pour into the bottle 5 cc. of clear limewater; then quickly replace the glass plate and, holding it firmly against the mouth of the bottle, shake the contents of the bottle. Note any change in the appearance of the limewater. Now tip the bottle as far as possible without spilling the limewater; remove the glass plate and quickly ignite the gas, holding the bottle in this position so that at least a portion of the combustion product may be retained in the bottle. When the flame dies out, at once cover the mouth of the bottle with the glass plate and shake the contents. (7) Record and explain the results.

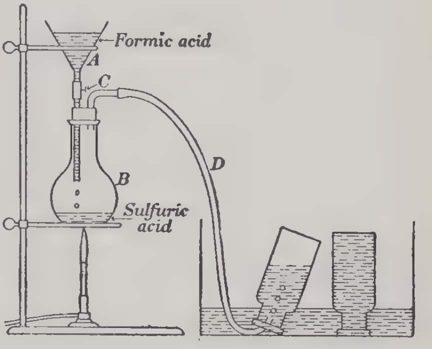


FIG. 49

Introduce into the tube *A* (Fig. 50) a small quantity of copper oxide and arrange the apparatus as shown in the figure. The bottle *C* contains limewater. Now connect the exit tube *D* (Fig. 49) with the tube *A* (Fig. 50). Heat the copper oxide gently, moving the flame so as to heat the oxide uniformly. When the oxide is heated to redness or nearly so, pass a slow current of carbon monoxide through the tube, generating the gas as under experiment *a*. The temperature of the copper oxide must be maintained by constant heating. Continue until both the limewater and the copper oxide have visibly changed. (8) Describe the results and write the equations for all the reactions involved.

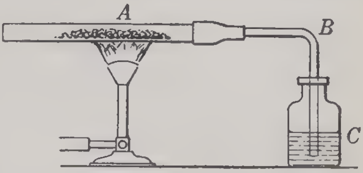


FIG. 50

*c.* (9) Summarize the properties and chemical conduct of carbon monoxide.

## EXERCISE 26

## THE QUANTITATIVE COMPOSITION OF CARBON DIOXIDE

**Object.** To determine the composition of carbon dioxide quantitatively and to deduce the percentage composition of carbon dioxide; also to calculate the combining weight of carbon, and the simplest formula of carbon dioxide.

**Apparatus.** An apparatus as shown in Fig. 51. *A* is a large beaker and *B* is a bottle having a capacity of at least 1 liter. The U-tube *L* should be a 4-in. tube. The tubes *D*, *H*, and *L* are filled with reagents, as designated in the figure. The glass tubes used in the connections are the ordinary glass tubes contained in your locker outfit except the tube *M*, which is a 1-mm. capillary tube (obtain from instructor). The rate of the oxygen flow is controlled by the capillary tube, and the screw clamp *C* is used only as a shut-off. Four short glass tubes sealed at one end will be needed to close the pure gum tubes *K*, *I*, and *G* when the tubes *L* and *H* are disconnected for weighing. There will also be required the apparatus used for preparing oxygen (Fig. 29).

**Materials.** Granular soda lime and calcium chloride sufficient to fill the tubes *D*, *H*, and *L* as designated in the figure (the calcium chloride in tube *H* must have been previously saturated with carbon dioxide), — the granules should have an average diameter of from 1.5 to 2 mm. and should be *loosely* packed in the tubes; 10 g. of potassium chlorate and 4 g. of manganese dioxide for preparing oxygen; 10 g. of charcoal (small lumps).

*a. Preparatory steps.* Fill the bottle *B* (Fig. 51) with oxygen prepared according to the methods given in Exercise 6, Fig. 29. (If compressed oxygen in steel cylinders is available, this may be utilized, *but the compressed oxygen must not be withdrawn from the cylinders except under the supervision of the instructor.*)

Place the charcoal in the tube *F* and weigh tube and contents to 0.01 g. Insert all weights in their proper places in the table on page 56. The tubes *L* and *H*, filled with soda lime and calcium chloride as shown in the figure, are made air-tight by inserting the glass seals; they are weighed to 0.01 g. and the weights are recorded in the table.

Now connect the apparatus as shown in the figure. The glass seals used for making the tubes *L* and *H* air-tight while weighing should be carefully laid aside since they must be inserted again when the tubes are reweighed. Care must be taken that the various stoppers fit the tubes snugly so that the apparatus will be air-tight except at the opening *K*. As a precautionary measure fold a wire gauze into a hollow cylinder of such a diameter as is needed to fit the gauze snugly about the tube *F*. The siphon tube *N*, leading from the beaker *A* into the bottle *B*, is filled with water and tightly closed by the clamp *C*.

*b.* Having the weights of the tubes *F*, *H*, and *L*, and having the apparatus all carefully connected as shown in the figure, we are now ready to proceed with the synthesis of the carbon dioxide. To do this, cautiously heat the wire gauze about the tube *F* at the point nearest the charcoal. After a few minutes, admit the water into the bottle *B* by opening the clamp *C*, thus forcing a slow current of oxygen over the heated carbon. The end of the siphon tube *N* must always be kept below the water in *A* while the combustion of the carbon is in progress. The moisture is removed from the oxygen as the current of gas flows through the tube *D*. Any moisture in the charcoal will be expelled by the heat and absorbed in tube *H*; hence the gain in weight in tube *H* must be subtracted from the original weight of the charcoal in order to obtain the weight of the pure charcoal. The charcoal will glow when the pure oxygen comes in contact with it. Continue the experiment until about 1 liter of oxygen has been forced out of the bottle *B*; then withdraw the flame from the tube *F* so that it may cool as rapidly as possible. Now disconnect the bottle *B* and entirely fill it with water (if not already filled) so as to drive out any remaining oxygen in the bottle. Then empty it, connect it again with the apparatus, and force about 500 cc. of air through the apparatus so as to replace the oxygen in the tubes with air. After the tube *F* has cooled to room temperature, disconnect the tubes *F*, *H*, and *L*, inserting the glass seals in the tubes *L* and *H*, so as to render them air-tight. Now weigh each of the tubes *F*, *H*, and *L* and contents to 0.01 g. Fill in the following table with the appropriate experimental data and make the necessary calculations.

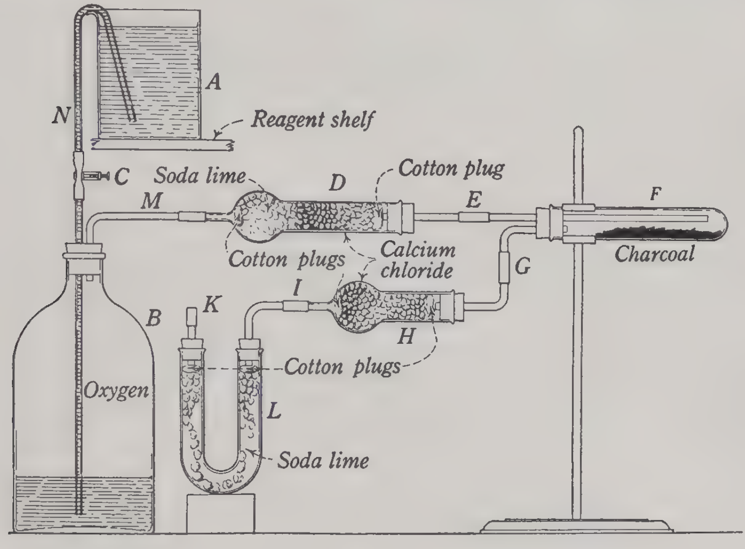


FIG. 51

### Table of Results

1. Weight of tube $H$ and contents (before absorption of moisture) . . . g.
2. Weight of tube $H$ and contents (after absorption of moisture) . . . . g.
3. Weight of moisture expelled from charcoal (Calculate) . . . . . . . g.
4. Weight of tube $F$ and contents (before heating) . . . . . . . . . . g.
5. Weight of tube $F$ and contents (after heating) . . . . . . . . . . . g.
6. Weight of carbon oxidized to carbon dioxide (equals loss in weight of tube $F$ and contents upon heating minus increase in weight of tube $H$ and contents after absorption of moisture) . . . . . . . . . . g.
7. Weight of tube $L$ and contents (after absorption of carbon dioxide) . g.
8. Weight of tube $L$ and contents (before absorption of carbon dioxide) . g.
9. Weight of carbon dioxide formed (Calculate) . . . . . . . . . . . g.
10. Weight of oxygen used (Calculate) . . . . . . . . . . . . . . . . . g.

*c. Calculations.* From the weights of the carbon and oxygen used and that of the carbon dioxide formed in this experiment, make the following calculations: (1) the percentage composition of carbon dioxide; (2) the combining weight of carbon; (3) the simplest formula for carbon dioxide.

## EXERCISE 27

## THE WEIGHT OF A DEFINITE VOLUME OF CARBON DIOXIDE; THE MOLECULAR FORMULA OF CARBON DIOXIDE

**Object.** To determine the weight of 1 liter of carbon dioxide and to calculate from this the molecular weight of carbon dioxide.

**Apparatus.** General apparatus arranged as shown in Fig. 52 (see below for description); apparatus for generating carbon dioxide according to Fig. 48.

**Materials.** Sufficient granular calcium chloride and soda lime to fill the tubes $I$ and $J$ as described below; 5 pieces of marble the size of walnuts for generating carbon dioxide according to Exercise 24; hydrochloric acid; sulfuric acid.

***a.*** Arrange an apparatus like that shown in Fig. 52. $C$ is an iron (or wooden) rod attached to a ring stand by a universal clamp $B$. A part of each of the tubes $I$ and $J$ is filled with soda lime and the remaining part with calcium chloride, as shown in the figure, loose cotton plugs being inserted at the four places marked $a$. The connections $M$, $N$, and $N'$ are pure gum tubing; $D$ and $F$ may be ordinary rubber tubing. Glass plugs made of short glass tubes sealed at one end are needed to close the rubber connections $N$ and $N'$ on the U-tube $J$ when it is detached from the remainder of the apparatus. Since the apparatus consists of a number of parts and it is essential that it should be airtight, it is well to connect the parts as shown in the figure and to make a preliminary test to see that there is no leak. To do this, fill the bottle $E$ with tap water, start the siphon exit tube $F$, then close the screw clamp $G$. Now connect the parts of the apparatus. Close the free end of the drying tube $I$ by slipping over the tube $S$ a piece of pure gum

tubing made air-tight by a screw clamp or a glass plug. Now open the screw clamp *G*. If the apparatus is air-tight there will be a slight flow of water at first, after which it will cease. If the water continues to flow, the faulty connection must be located. To do this, disconnect the apparatus in turn at the various points of connection, beginning with *D*. Seal the rubber tube *D* with a glass seal or a screw clamp so as to make it air-tight; then open the clamp *G* and see if the water continues to flow. If it does, the stopper in the bottle and the tubes must be adjusted until the apparatus is air-tight. Now connect the apparatus again at *D*. Then in a similar way test each of the other points of connection until you have found the faulty places and made them air-tight.

Remove the tube *J* from the apparatus, being sure to keep the connections *N* and *N′* on the tube. Close each of these with the sealed glass plugs and carefully weigh the tube and connections to 0.01 g., inserting the weight in the appropriate place in the table (page 58). Set the tube aside temporarily.

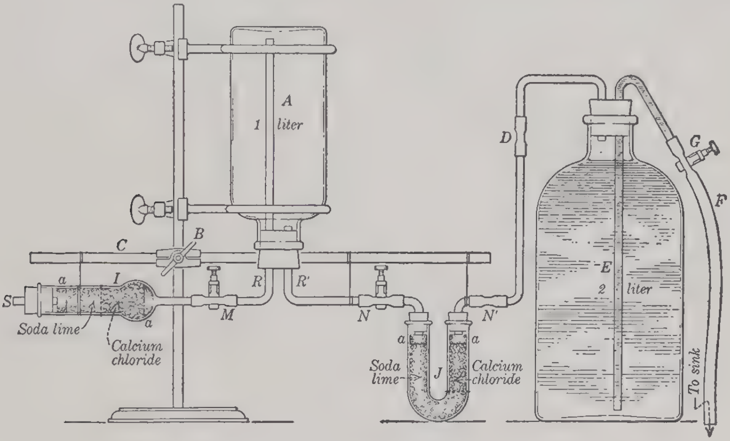


FIG. 52

Now disconnect the bottle *A* from the tube *I*. Remove the stopper with its attached bent tubes. Dry the bottle with an air blast. Then place it in an upright position and insert the rubber stopper and tubes. Next fill the bottle *A* through the tube *R* with carbon dioxide prepared as described in Exercise 24 and dried by causing it to bubble slowly through some sulfuric acid. (If compressed carbon dioxide is available, this may be used, *but the compressed gas must not be withdrawn except under the supervision of the instructor*.) To tell when the bottle is filled, test the gas escaping from the tube *R′* with a lighted splint. The carbon dioxide should be allowed to flow in until there is no doubt that the bottle is filled. When the bottle is filled, close the connections *M* and *N* with pinch clamps.

Now connect the apparatus as shown in Fig. 52. Be careful to save the two glass plugs used in closing the tubes $N$ and $N'$ so that these may be inserted again when the tube is to be reweighed. Be careful to leave the rubber tubes $M$ and $N$ sealed with screw clamps. Now mark, by a label, the depth to which the stopper extends into the bottle $A$. Open the screw clamps from the rubber connections $M$ and $N$ and *at once* partly open the screw clamp $G$ so that a slow stream of water escapes through $F$. As the water flows out of the bottle $E$, the carbon dioxide in the bottle $A$ is drawn out and is absorbed by the contents of the tube $J$. If the experiment is to be successful, all the carbon dioxide must be absorbed; hence the importance of allowing the water to flow *slowly* from the bottle $E$. The absorption of the carbon dioxide in the tube $J$ evolves heat; if the tube becomes hot to your hand, stop the flow of the gas from $A$ until the tube becomes cool. After the bottle $E$ is empty, disconnect the tube $J$, inserting the glass plugs set aside for this purpose, and reweigh. Insert the weight in the appropriate place in the table below. This gain in weight is the weight of the carbon dioxide originally contained in bottle $A$. The volume of the carbon dioxide is determined by measuring the amount of water in $A$ when it is filled to the mark made by the gummed label. The stopper and its bent glass tubes must be kept in the bottle $A$ until the water reaches the mark. Insert the value in the appropriate place in the table below. Now obtain the temperature of the room and the barometric pressure and insert the figures in the appropriate places in the table below. From these results make the calculations required and insert in the appropriate places.

TABLE OF RESULTS

| | |
|---|---|
| 1. Weight of tube $J$ + carbon dioxide | g. |
| 2. Weight of tube $J$ | g. |
| 3. Weight of carbon dioxide (Calculate) | g. |
| 4. Barometric pressure | mm. |
| 5. Temperature of room | ° |
| 6. Volume of carbon dioxide at atmospheric pressure | cc. |
| 7. Volume of the carbon dioxide under standard conditions (Calculate) | cc. |

*b. Calculations.* From the above data and those obtained in the synthesis of carbon dioxide (see Exercise 26), make the following calculations: (1) weight of 1 liter of carbon dioxide under standard conditions; (2) the molecular weight of carbon dioxide; (3) the molecular formula for carbon dioxide.

## EXERCISE 28

## NITROGEN

**Object.** To prepare nitrogen and to study its properties.

**Apparatus.** Apparatus required for preparing nitrogen by method 1: Apparatus shown in Fig. 53. *A* is the *hard*-glass tube included in your outfit and is supported on a ring stand; it contains a spiral of fine copper gauze (from 60 to 70 mesh), made by rolling up a piece of gauze about 10 × 12 cm.; the gauze must fit the tube loosely so that it can be pushed easily to the middle of the tube; *B* is a 1-liter (or larger) bottle (the ordinary 5-pint acid bottle serves the purpose well); the glass tube *D* extends to the bottom of the bottle *B* and is connected to a water tap by means of a rubber tube); 3 wide-mouthed bottles (250-cc.); pneumatic trough; wing-top burner; 3 glass plates to fit over the bottles when removed from the trough.

Apparatus required for preparing nitrogen by method 2: pneumatic trough; porcelain crucible; wide-mouthed bottle (the mouth must be wider than the crucible); file or piece of wire; glass plate.

Apparatus required for preparing nitrogen by method 3: 250-cc. flask, with cork and glass delivery tube like that used in preparing oxygen (*B*, *C*, Fig. 29); ring stand; burner; pneumatic trough; 3 wide-mouthed bottles and glass plates.

**Materials.** For method 1: piece of copper gauze (from 60 to 70 mesh) 10 × 12 cm.; splint.

For method 2: small piece of phosphorus to be obtained from the instructor when needed; splint.

For method 3: 3 g. of ammonium chloride; 6 g. of sodium nitrite; splint.

NOTE. Three methods for preparing nitrogen are given below. The method for preparing it from air by the action of copper, while requiring more time, has an advantage, since the chemical changes involved are simple and are easily observed through the change of the reddish copper into the black copper oxide.

*a. Preparation of nitrogen from the air by the action of copper (method 1).* When air is passed through a tube containing hot copper, the oxygen in the air combines with the copper, leaving the nitrogen. This gives a simple method for preparing nitrogen. Proceed as follows: Arrange the apparatus as shown in Fig. 53, except that the end of the exit tube *F* is not placed in the mouth of the bottle *C*, and see that the parts are all well connected. The bottle *B* is empty at the beginning, while the wide-mouthed bottles inverted in *G* are completely filled with water. Now (gently at first) heat the copper spiral in the tube, using a wing-top burner, until the gauze is dull red; this will require about five minutes. Then continue the heating, bring the end of the tube *F* into the mouth of the bottle *C*, as shown in Fig. 53, and at the same time admit a *slow* current of water into *B* at a rate not to exceed 1 liter in twenty minutes. The water entering *B* forces the air in the bottle over the hot copper, which unites with the oxygen, while the nitrogen passes on and is collected in the bottles in *G*. When two of these bottles are filled, disconnect the apparatus and withdraw the heat. (1) Why disconnect the apparatus before withdrawing the heat? (2) Note and explain the change in color of the copper spiral.

Remove the bottles from *G* as in the preparation of oxygen and hydrogen, keeping each covered with a glass plate. Since the copper removes only the oxygen, the nitrogen prepared by this method will contain small percentages of the rare elements present in the atmosphere. Since they are present in such small quantities (about 1 per cent of the original air), and since they resemble nitrogen so closely, they do not materially affect the properties of the nitrogen.

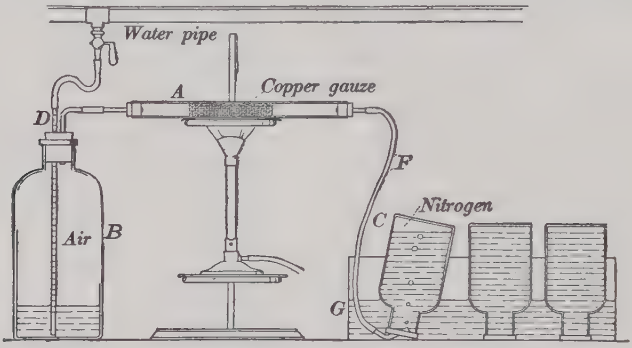


FIG. 53

***Caution.*** *On account of its great affinity for oxygen, phosphorus must be kept and handled under water. Never bring the dry substance in contact with the skin, as it may ignite and cause a serious burn.*

***b.*** *Preparation of nitrogen from the air by the action of phosphorus* (*method 2, optional*). Cover the bottom of a pneumatic trough with water to a depth of 2 or 3 cm. Float on the water a porcelain crucible containing a small piece of phosphorus. Ignite the phosphorus by touching it with the hot end of a wire or file (Fig. 31), and quickly invert over the crucible a large wide-mouthed bottle or beaker, being careful to keep the mouth of the bottle below the surface of the water. The white fumes formed consist of the finely divided white solid compound of an oxide of phosphorus, which is gradually dissolved in the water. Leave the bottle in position until the fumes have entirely disappeared. Note that the water has risen in the bottle. (3) Explain. Adjust the bottle or the water in the trough so that the level of the liquid inside and outside of the bottle is the same; then cover the bottle with a glass plate and set it in an upright position on the desk.

***c.*** *Preparation of nitrogen from ammonium nitrite* (*method 3, optional*). Since ammonium nitrite is not easily obtained in a pure state, it is

convenient to use a mixture of the two more available compounds, ammonium chloride and sodium nitrite. When heated together these react to form ammonium nitrite, which then decomposes into nitrogen and water (page 179 of text). To prepare nitrogen from these, proceed as follows:

In a 250-cc. flask introduce a mixture of 3 g. of ammonium chloride and 6 g. of sodium nitrite, and add 20 cc. of water. Provide the flask with a one-hole cork and delivery tube so that the evolved gas may be collected over water, as in the case of oxygen and hydrogen. Have at hand a vessel of cold water so that the flask may be cooled by immersing it in the water in case the action becomes too violent.

Clamp the flask and apply a *very gentle* heat, moving the burner about with the hand. As soon as the action begins, withdraw the heat. After the air has been expelled from the apparatus, fill three bottles (250-cc.) with the gas. If the action becomes too violent, immerse the flask in cold water. (4) Which of the three methods for preparing nitrogen, given above, gives the purest product?

*d. Properties and chemical conduct of nitrogen.* (5) Note the color, odor, taste, and solubility in water of nitrogen. Insert a burning splint into a bottle of the gas. (6) Is nitrogen combustible? (7) Is it a supporter of combustion? (8) Summarize briefly the properties of nitrogen.

## EXERCISE 29

## THE COMPOSITION OF AIR

**Object.** To learn the composition of air and to determine quantitatively the percentage of oxygen and nitrogen present.

**Apparatus.** Apparatus shown in Fig. 54 or Fig. 55, depending on the method selected (the apparatus shown in Fig. 54 is the same as that shown in Fig. 53, and described under Exercise 28, except that the bottle *B* holds not less than 2 liters, while bottle *C* holds about 1 liter); cork that fits bottle *C*. Graduated cylinder, beaker, 2 strips of gummed paper.

**Materials.** For method 1: copper gauze spiral like that used in Exercise 28.

For method 2: 5 g. of potassium hydroxide dissolved in 5 cc. of water and cooled to room temperature, and 4 g. of pyrogallic acid dissolved in 10 cc. of water.

*a.* Experiments already performed have proved the presence of certain constituents of the air. (1) Enumerate the experiments and their bearing on the composition of air.

*b. The relative volumes of oxygen and nitrogen in the air* (*method 1*). To determine the relative volumes of oxygen and nitrogen in air, a definite volume of air is passed *slowly* over hot copper, which combines with the oxygen. The remaining nitrogen is collected and its volume measured. The experiment is the same as in Exercise 28 except that the volume of the air passed over the copper gauze and that of the resulting nitrogen are both measured.

Arrange the apparatus as shown in Fig. 54. The glass tube $A$ contains the copper spiral. This spiral must be of such a size that it will just enter the tube and yet can easily be pushed to the middle part of the tube in the position shown in the figure. See that the corks and tubes are air-tight. At the beginning of the experiment bottle $B$ is empty, while bottle $C$ is *completely filled* with water and inverted in $G$. The end of tube $D$ must touch the bottom of bottle $B$.

When the apparatus is all connected, heat the copper spiral in tube $A$ with a wing-top burner until it is at a dull-red heat, and proceed to prepare nitrogen exactly as in *a*, Exercise 28. Care must be taken not to hasten the experiment, for in that case some of the oxygen may not combine with the copper. When the bottle $C$ is nearly full, turn off the water, withdraw the heat, and disconnect the apparatus.

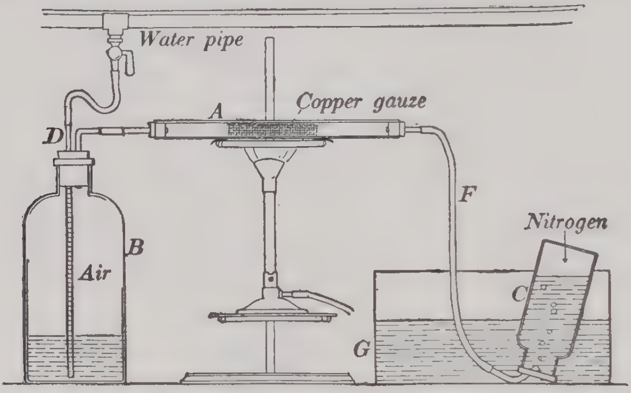


FIG. 54

Now raise (or lower) the bottle $C$ until the water is at the *same level* inside and outside the bottle (it may be necessary to add more water to the trough); then, while holding it in this position, push the cork tightly into the mouth of the bottle. Finally lift the bottle from the trough and place it in an upright position on your desk.

By means of a graduated cylinder measure the volume of the water in $B$; this equals the volume of air analyzed. Record the volume in the table on page 63. In the same way measure the volume of the nitrogen in $C$ (this contains about 1 per cent of rare gases, chiefly argon) and record it in the table. Then from your results calculate the volumes of nitrogen and oxygen in 100 volumes of air, recalling that 100 volumes of air contains approximately 1 volume of the rare gases which remain mixed with the nitrogen. (2) Record your results in the appropriate places in the table on the following page. (3) Could silver be substituted for copper in the above experiment?

### TABLE OF RESULTS

1. Volume of air analyzed (volume of water in $B$) . . . . . cc.
2. Volume of nitrogen and rare gases in the volume of air analyzed (volume of gas in $C$) . . . . . . . . . . . . . cc.
3. Volume of oxygen in volume of air analyzed (difference between volumes 1 and 2) . . . . . . . . . . . . . . . . cc.
4. Volume of nitrogen and rare gases in 100 volumes of air (Calculate) . . . . . . . . . . . . . . . . . . . . . . cc.
5. Volume of nitrogen alone in 100 volumes of air (equals volume of nitrogen and rare gases in 100 volumes of air less 1 volume) . . . . . . . . . . . . . . . . . . . . . . . cc.
6. Volume of oxygen in 100 volumes of air (Calculate) . . . . cc.

| | ACTUAL | RESULTS OBTAINED | AVERAGE OF RESULTS OBTAINED BY CLASS |
|---|---|---|---|
| 7. Volume of oxygen in 100 volumes of air . | 21 | | |
| 8. Volume of nitrogen in 100 volumes of air . | 78 | | |

(4) What are the sources of error in the above experiment?

*c. The relative volumes of oxygen and nitrogen in the air* (*method 2*). The relative volumes of oxygen and nitrogen in the air may be determined by bringing in contact with a definite volume of air a liquid that absorbs the oxygen and in so doing flows into the tube that contains the air, and fills a space equal to that previously occupied by the oxygen. The volume of this liquid is then measured and the volume of the absorbed oxygen ascertained.

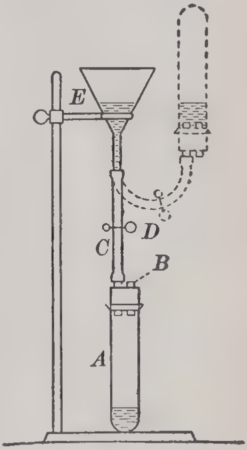


FIG. 55

The solution used to absorb the oxygen soon loses its strength on exposure to air; the experiment should therefore be performed rapidly. Before preparing the solution the student should practice the manipulations involved in the experiment, as skill is required for accuracy.

Arrange an apparatus like that shown in Fig. 55. $A$ represents a test tube about 15 cm. in length (use the hard-glass tube employed in the preparation of oxygen). The tube is fitted with a two-hole rubber stopper. One hole is closed with a glass rod $B$, while the other is fitted with a small glass tube, the upper end of which extends 4 or 5 cm. above the stopper. A piece of soft rubber tubing $C$, 12 or 15 cm. in length, connects the glass tube with a small funnel, as shown in the figure. Have at hand a small beaker and 2 strips of gummed paper, 1 or 2 cm. in length.

Close the rubber tube tightly with the screw clamp $D$. Disconnect the test tube and remove the glass rod $B$ from the stopper preparatory to performing the experiment.

Add the solution of potassium hydroxide to the solution of pyrogallic acid (see "Materials"), mix, and *at once* pour the resulting liquid into the funnel *E*. Place a small beaker under the tube *C* and quickly open the screw clamp *D* until the rubber tube and the glass tube are both filled with the liquid; then close the clamp tightly.

Connect the test tube *A* to the tube *C* by inserting the stopper, holding it by the rim to avoid heating the contained air, and insert the glass rod *B* in the cork. The air inclosed in the tube is now at the same temperature and pressure as the surrounding air.

Now open the screw clamp *D*. The liquid flows into *A*, absorbing the oxygen. When the liquid ceases to enter, grasp the tube by the rim and invert it, as shown by the dotted lines of the figure, adjusting it so that the level of the liquid is the same in both tube and funnel; then clamp the rubber tube tightly and return the test tube to its original position.

Mark the volume of the air originally inclosed in the tube by placing a narrow strip of gummed paper about the tube at the lower end of the stopper; mark also, by a strip of paper placed at the level of the liquid in the tube, the volume of the oxygen absorbed.

Disconnect the tube and rinse it. Measure the volume of the tube to each strip of paper by pouring in water from a graduated cylinder. From these measurements calculate the number of volumes of oxygen and nitrogen in 100 volumes of air. (5) Insert the values in the following table:

### TABLE OF RESULTS

1. Volume of air analyzed (volume of tube *A* to lower end of stopper) . . . . . . . . . . . . . . . . . . . . . . cc.
2. Volume of oxygen present (volume of liquid in *A*) . . . . cc.
3. Volume of oxygen present in 100 volumes of air (Calculate) cc.
4. Volume of nitrogen and rare gases in 100 volumes of air (Calculate) . . . . . . . . . . . . . . . . . . . . . . cc.
5. Volume of nitrogen in 100 volumes of air (equals volume of nitrogen and rare gases in 100 volumes of air less 1 volume) cc.

## EXERCISE 30

## SOME FURTHER MANIPULATIONS

**Object.** To learn how to transfer a small volume of a liquid.

**Apparatus.** File; forceps; medicine dropper; graduated pipette; burner; test tube; beaker.

**Materials.** Glass tubing, 25 cm. in length and 3 or 4 mm. inside diameter.

*Devices for transferring liquids drop by drop.* An ordinary medicine dropper (obtainable at any drug store) is useful for transferring a few drops of a liquid from one vessel to another (Fig. 56). A glass tube made as described below is often of more service. It is known as a *pipette.*

Select a piece of glass tubing about 25 cm. long. Heat the glass tube, about 5 cm. from one end, in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (*A*, Fig. 57). The tube must be constantly rotated to prevent the softened portion from sagging. Now quickly remove the tube from the flame and, holding it in a vertical position, gently pull the ends apart until the bore is of the desired size (*B*, Fig. 57). The tube is then cut at *B*, and both ends of the longer piece are rounded by heating in the flame as directed in Exercise 2 (Fig. 11).

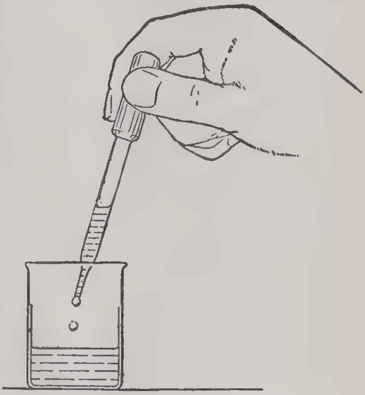


FIG. 56

To transfer a liquid from one vessel into another, dip the tip of the pipette below the surface of the liquid to be transferred and, placing the other end in the mouth, gently suck the liquid up in the *pipette* until partly filled. *Great care must be taken to keep the tip of the pipette well below the surface of the liquid while it is being drawn up. Moreover, the pipette should never be filled more than about two thirds full; otherwise the liquid may be drawn into the mouth and serious results follow.* When the pipette is partly filled, remove the end from your mouth and quickly press your forefinger firmly on it. The liquid

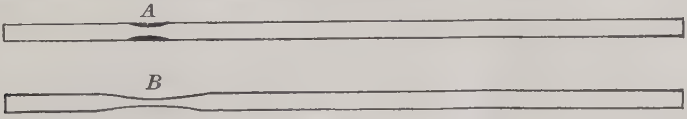


FIG. 57

in the pipette may now be transferred to another vessel by bringing the tip over the mouth of the vessel and then slightly decreasing the pressure of the finger upon the end of the pipette, as shown in Fig. 58.

A *graduated pipette* is a tube such as just described, but graduated in cubic centimeters. It serves the same purpose, but has the additional advantage that it enables one to tell just how much of the liquid is transferred. In using such a pipette the liquid is drawn up a little above the zero point. The finger is then lightly pressed against the end of the pipette, and the liquid is allowed to flow out until the level of the liquid is just at the zero mark, when the flow is stopped by increasing the pressure of the finger against the end of the pipette. The liquid may then be allowed to drop (or flow) into any vessel until the desired amount is transferred. The flow is stopped by increasing the pressure of the finger on the end of the pipette.

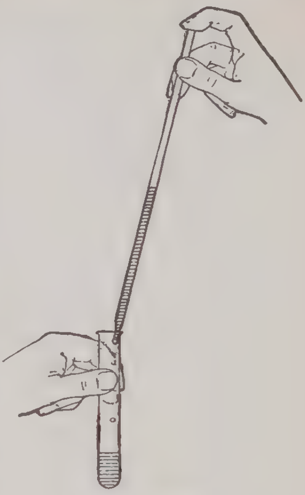


FIG. 58

# EXERCISE 31

## SOLUTIONS

**Object.** To study the solubilities of various solids and to learn something of the properties of solutions.

**Apparatus.** Test tubes; graduated pipette; funnel and filter paper; 100-cc. beaker; watch glass; ring stand and burner; apparatus shown in Fig. 44 (*A* is a 250-cc. flask, *B* is a thermometer, and *C* is an open glass tube).

**Materials.** 2 crystals of potassium permanganate; 0.2 g. of powdered calcium sulfate; 5 g. of common salt; 2 g. of potassium nitrate; 1 g. of sugar; 10 cc. of carbon tetrachloride (R.S.); 1 cc. of oil (cottonseed or olive); 10 g. of sodium thiosulfate.

*a.* Nearly fill two test tubes with water and set them in a rack. Drop into each a small crystal of potassium permanganate. Shake the contents of one tube and repeat from time to time, but do not move the other tube. At the close of the laboratory period note the appearance of the liquid in each tube. (1) Record your conclusions.

*b.* Introduce about 0.2 g. of finely powdered calcium sulfate into a test tube and add 5 cc. of distilled water. Shake the mixture thoroughly and set it aside for ten minutes. (2) Is there any evidence that the solid is soluble in water? Now filter a few drops of the liquid, collecting the filtrate on a watch glass. Set the glass on a small beaker half full of boiling water (Fig. 59) until the liquid is evaporated. (3) What conclusions do you draw with reference to the solubility of calcium sulfate?

*c.* Place exactly 2 g. of common salt in one test tube and an equal weight of potassium nitrate in another. Add to each exactly 2 cc. of water (use a graduated pipette) and heat each tube until the water boils. If the solid does not dissolve, add an additional cubic centimeter of water and again heat. Repeat until the solid in each tube is dissolved. (4) Compare the solubilities of the two solids in the boiling water.

Cool the solutions in each tube and note the approximate quantities of solids separating. (5) State your results. (6) Are they in general accord with the solubilities of the two solids given on page 239 of text?

*d.* Introduce about 0.5 g. of sugar into each of two test tubes. To the one tube add 5 cc. of water and to the other 5 cc. of carbon tetrachloride. (This is a colorless, noninflammable liquid and has the formula $CCl_4$.) Shake the tubes gently and note the results. Repeat the experiment, substituting a small bit of lard or some oil for the sugar. (7) How could you remove stains on cloth, caused by a sugar sirup (molasses)? by oils or grease? by a mixture of the two?

*e.* Introduce into a test tube 10 g. of sodium thiosulfate (the ordinary "hypo" of the photographer) and add 2 cc. of water. Heat the tube gently until a uniform solution is obtained, taking care that no particles of the solid remain on the side of the tube; stopper the tube loosely with a plug of cotton and set it aside until the solution is cold. If sufficient care has been taken, no solid will have separated. Now remove the cotton plug and drop into the solution a bit of the solid "hypo" just large enough to see. (8) Note the results (hold the tube in a good light) and explain. Bring your hand in contact with the test tube as the solid is separating and notice whether there is any appreciable change of temperature. (9) Explain.

*f*. Prepare about 25 cc. of a saturated solution of sodium chloride and determine its boiling point (Fig. 44). (10) Explain.

## EXERCISE 32

## THE SOLUBILITY OF CERTAIN SOLIDS IN WATER

**Object.** To determine the solubility of certain solids in water at different temperatures.

**Apparatus.** Hard-glass test tube used in preparing oxygen, with cork stopper to fit; large beaker; thermometer registering to at least 100°; small evaporating dish and glass cover; funnel and filter to fit; ring stand; burner.

**Materials.** 15 to 20 g. of sodium chloride, potassium chloride, or potassium dichromate; pieces of ice.

NOTE. It is suggested that certain students be given the problem of determining the solubility of a solid in water at 0°, others at 20°, etc., so that the solubility of the solid may thus be determined at the temperatures 0°, 20°, 40°, 60°, 80°, and 100°. These results may then be collected and each student will be able to construct a solubility curve by means of the diagram (Fig. 60).

*a*. Introduce about 10 g. of the *finely powdered* solid chosen (sodium chloride, potassium chloride, or potassium dichromate) into a rather large test tube (the hard-glass test tube used in preparing oxygen serves well); add about 25 cc. of water to this and loosely stopper it with a cork. Nearly fill a large-sized beaker with water, place a thermometer in this, and cool or heat the water to the desired temperature, maintaining this as nearly as possible. Now thoroughly mix the solid and water in the test tube by shaking it, and then place the tube in the beaker of water so that the mixture in the tube is entirely immersed in the water. The tube should be left in the water until a saturated solution of the solid at the desired temperature has been formed. The time required will vary with the solid but should be complete in ten minutes. The tube should be withdrawn from the water from time to time, long enough to shake the contents and thus hasten the solution. If the solid should all or nearly all dissolve, more must be added; otherwise the solution formed will not be saturated.

While the solid is dissolving, accurately weigh a small evaporating dish and watch-glass cover to 0.01 g. and record the weights in the table at the bottom of the page. Next fit a filter paper into a dry funnel and arrange it so that the liquid to be filtered will flow into the weighed evaporating dish. For filtering the solutions made at a higher temperature than 40° it is necessary to have the funnel hot in order to prevent the solid from separating from the solution in the cold stem of the funnel. To do this dip it in boiling water until it has acquired the temperature of the water; then quickly dry it and proceed at once with the filtration as directed below. After a saturated solution of the solid has been formed, quickly filter it into the weighed evaporating dish, cover the dish with the watch glass, and set it aside until the dish and contents are at approximately room temperature. Then reweigh the dish, cover, and solution, recording the weight in the table. Next remove the watch glass and place the dish on a beaker partially filled with water as shown in Fig. 59. Keep the water in the beaker boiling. After the solution in the dish has evaporated to dryness, remove the dish and place it on a ring stand (Fig. 25). Now cover the dish with the watch glass and heat it directly with the burner, regulating the flame so that the tip barely touches the dish. Continue the heating until all the moisture has been expelled and the under part of the watch glass is free from moisture.

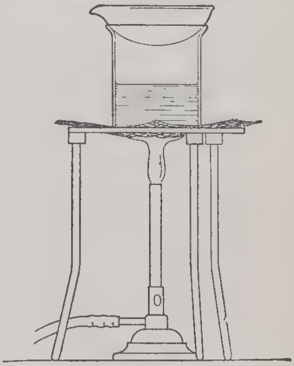


FIG. 59

Now withdraw the burner, and after the dish is cool (room temperature) weigh the dish, residue, and cover, recording the weight in the table below.

From the results so recorded, fill in the remaining blanks in the table and make the calculations called for. (1) Compare your results with those given on page 235 of text.

TABLE OF RESULTS

1. Solid selected . . . . . . . . . . . . . . . . . . . . . . . . . .
2. Temperature of the solution . . . . . . . . . . . . . . . . . . °
3. Weight of empty dish and glass cover . . . . . . . . . . . . g.
4. Weight of dish, solution, and glass cover . . . . . . . . . . g.
5. Weight of solution taken . . . . . . . . . . . . . . . . . . . . g.
6. Weight of dish, residue left after evaporation of water, and glass cover . . . . . . . . . . . . . . . . . . . . . . . . . g.
7. Weight of water present in solution (loss by evaporation) . . g.
8. Weight of solid dissolved in the water (residue) . . . . . . . g.
9. Weight of solid that will dissolve in 100 g. of water at the temperature of the solution (Calculate) . . . . . . . . . . . g.

*b.* Collect from other students the solubilities of the solid at different temperatures and record them below:

Solubility of ____________________ expressed in grams per 100 g. of water, at 0°, ____; at 20°, ____; at 40°, ____; at 60°, ____; at 80°, ____; at 100°, ____.

Solubility Curve

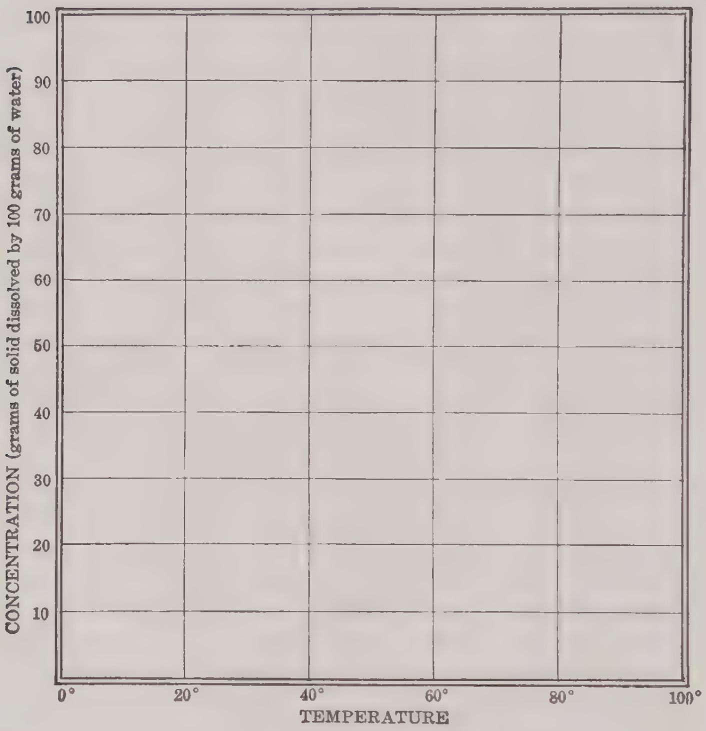


Fig. 60

From these results draw the solubility curve on the above diagram (Fig. 60) (consult the diagram on page 235 of text).

# EXERCISE 33

## CHLORINE

**Object.** To prepare chlorine and to study its properties.

**Apparatus.** Apparatus as shown in Fig. 61 (*A* is a 250-cc. flask and *B* and *C* are 250-cc. bottles; *B* contains some sulfuric acid); 4 additional 250-cc. bottles (dry); glass plates; 200-cc. beaker with glass-plate cover.

**Materials.** 25 g. of manganese dioxide; hydrochloric acid; bit of powdered antimony; strip of copper foil; strips of colored calico; piece of printed paper (printer's ink); paper written over with ordinary ink; 1 g. of potassium permanganate.

***Caution.*** *Experiments **a**, **b**, and **c** must be performed in the hood, and great care must be taken not to inhale the chlorine, as the gas is very irritating and is poisonous.*

***a.*** *Preparation of chlorine (usual laboratory method).* (Two students may work together.) Arrange an apparatus according to Fig. 61. Put into the flask *A* from 20 to 25 g. of manganese dioxide. Insert the cork and pour 150 cc. of hydrochloric acid through the funnel tube. Shake the flask so as to mix the contents thoroughly. Warm gently, applying just enough heat to cause a gentle evolution of the gas but not enough to boil the liquid. The chlorine that is generated bubbles through the sulfuric acid in *B* (which removes the moisture) and is collected in *C*. Fill four bottles with the gas (the color of the gas will indicate when the bottle is filled), cover them with glass plates, and set them aside.

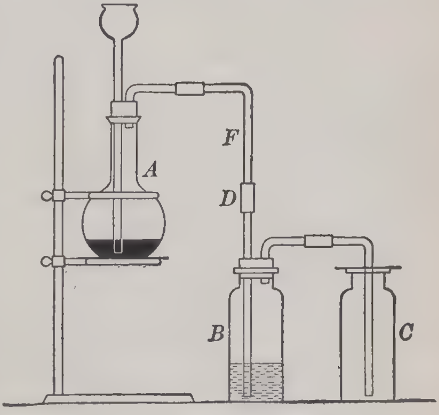


FIG. 61

(1) Write the equation for the reaction involved in the preparation. (The manganese (quadrivalent) of the manganese dioxide and the hydrogen of the acid exchange places, forming manganese tetrachloride ($MnCl_4$), which then breaks down into $MnCl_2$ and $Cl_2$.) Save the mixture in the flask *A* and, when time is available, recover the $MnCl_2$. (2) Describe the method used.

***b.*** *Properties of chlorine.* (3) Note the color of the gas. (4) Why is it not collected over water, like oxygen and hydrogen? (5) Give its solubility in water (page 250 of text). Allow a *small* amount of the gas to escape into the air and *cautiously* (6) note its odor.

*c. Chemical conduct of chlorine.* Grind a fragment of antimony to a fine powder and sprinkle a pinch of the powder into one of the bottles of the gas. Antimony trichloride ($SbCl_3$) is formed. (7) Write the equation for its formation.

Support by forceps a small piece of copper foil, heat it to redness, and *immediately* drop it into a bottle of the gas. (8) Describe the result. (9) What is formed?

Suspend strips of colored calico in a bottle of the *dry* gas; also two strips of paper, the one with writing in ink on it, the other with printing (printer's ink) on it. In another bottle of the gas suspend similar strips moistened with water. Allow them to remain for five or ten minutes. (10) Describe the results. (11) What part does the water play in the bleaching?

Place a few crystals of potassium permanganate on the bottom of a clean beaker. Pour over these 3 cc. of hydrochloric acid and cover the beaker with a glass plate. Examine after a few minutes and test the gas in the beaker for the presence of chlorine. (12) Note your conclusions.

(13) Summarize the properties and chemical conduct of chlorine.

## EXERCISE 34

### HYDROGEN CHLORIDE; HYDROCHLORIC ACID

**Object.** To prepare hydrogen chloride and hydrochloric acid and to study their properties.

**Apparatus.** Flask and bottle connected as shown in Fig. 62 (this is the same as shown in Fig. 61 except that the glass tube extending into the bottle *B* does not touch the liquid (water) in *B*); two 250-cc. bottles (dry); burner; large beaker; glass plates.

**Materials.** Dilute sulfuric acid prepared by *slowly* pouring (3 or 4 drops at a time with constant stirring and cooling) 30 cc. of the concentrated acid into 10 cc. of water; 50 g. of sodium chloride; splint; blue litmus paper.

*a. Preparation of hydrogen chloride and hydrochloric acid (usual laboratory method of preparation).* Put about 50 g. of common salt into the generator flask *A* (Fig. 62), insert the cork, pour the cold dilute sulfuric acid through the funnel tube, mix the contents by a gentle motion of the flask, and after two or three minutes warm gently with a *small* flame. Notice the currents in the water in *B*. (1) What causes them? As soon as the gas is evolved regularly, disconnect the generator flask at *D* long enough to collect two bottles of the gas by displacement of air (as in Fig. 48). Cover these tightly with glass plates and set them aside; then connect the generator with *B* again and continue to apply a gentle heat as long as any gas is evolved. (2) Write the equation for the reaction involved (the sodium of the sodium chloride and the hydrogen of the sulfuric acid simply exchange places).

*b. Properties of hydrogen chloride.* (3) Note the color and odor (*Care*) of the gas.

Fill a large beaker with water. Now invert one of the bottles of the gas over the beaker of water, remove the glass cover, and at once bring the mouth of the bottle under the surface of the water in the beaker. Hold it in this position four or five minutes, being careful to keep the mouth of the bottle always under the surface of the water. (4) Describe the results. (5) What does the experiment prove? Why not extend the tube in bottle *B* (Fig. 62) to the bottom of the bottle?

Test the gas in one of the bottles with a lighted splint. (6) Is it combustible or a supporter of combustion?

*c. Properties of hydrochloric acid.* Put a drop of the aqueous solution of the hydrogen chloride from bottle *B* (Fig. 62) on a bit of blue litmus paper. (7) Note the result. (8) Pour 2 drops of the solution into 3 or 4 cc. of water and taste a drop. Perform a test-tube experiment to prove the presence of chlorine in the acid; also one to prove the presence of hydrogen. (9) Describe the experiments and the results obtained. (10) How does the solution compare with the hydrochloric acid on your desk?

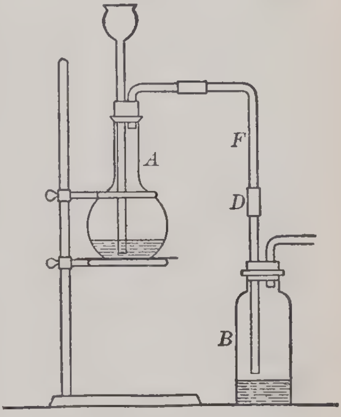


FIG. 62

(11) Distinguish clearly between hydrogen chloride and hydrochloric acid.

## EXERCISE 35

## SODIUM; SODIUM HYDROXIDE

**Object.** To study the properties of sodium and sodium hydroxide.
**Apparatus.** Evaporating dish with glass plate cover; forceps; glass rod.
**Materials.** Bit of sodium as large as a pea; red litmus paper.

*a. Properties of sodium.* Recall experiment *a*, Exercise 8. Obtain from your instructor a bit of sodium. Hold the sodium with the forceps on a glass plate, cut it, and note the rapidity with which the freshly cut surface is tarnished. Half fill your evaporating dish with water; then drop into this a bit of sodium as large as a pea, quickly cover the dish with the glass plate, and leave it covered until the sodium has all disappeared. (1) Is sodium heavier or lighter than water? (2) Write the equation for the reaction between sodium and water. (3) What is the composition of the liquid in the evaporating dish (page 51 of text)?

*b. Properties of sodium hydroxide.* Place a drop of the liquid in the evaporating dish on a piece of red litmus paper. (4) Contrast its action with that of hydrochloric acid on litmus paper (Exercise 34). Mix 1 or 2 drops of the solution with 5 cc. of water and taste a drop of the resulting solution. (5) Contrast with the taste of hydrochloric acid (Exercise 34). Evaporate the solution to dryness. (6) What is the residue?

## EXERCISE 36

## ACIDS, BASES, AND SALTS

**Object.** To study the composition and properties of acids, bases, and salts.

**Apparatus.** Small beaker; stirring rod; evaporating dish; ring stand; test tubes; medicine dropper.

**Materials.** A few drops of each of the following acids: hydrochloric, sulfuric, nitric, acetic; solutions of the following bases: sodium hydroxide, potassium hydroxide (R.S.), calcium hydroxide (R.S.); strips of blue and of red litmus paper; hydrochloric acid.

*a. Acids.* Recall the properties of hydrochloric acid (Exercise 34). Prepare a dilute solution of each of the following acids by adding 2 or 3 drops of the acid to about 10 cc. of water in a test tube and mixing thoroughly: hydrochloric ($HCl$), sulfuric ($H_2SO_4$), nitric ($HNO_3$), acetic ($HC_2H_3O_2$).

By means of a clean glass rod transfer a drop of each of the dilute solutions to a piece of blue litmus paper and also to a piece of red litmus paper. (1) Note the result in each case. (2) Taste *one* drop of the dilute solutions. (*Rinse the mouth with water after tasting.*)

Compare the formulas of the acids. (3) In what respect are the acids similar in composition?

*b. Bases.* (4) In a similar way try the effect on red and on blue litmus paper of a solution of each of the following bases: sodium hydroxide ($NaOH$) (recall Exercise 35), potassium hydroxide ($KOH$), calcium hydroxide ($Ca(OH)_2$). (5) Taste a drop of the calcium hydroxide solution.

Compare the formulas of the bases. (6) In what respect are the bases similar in composition?

*c. Salts.* Dilute 5 cc. of the ordinary laboratory solution of sodium hydroxide (120 g. of sodium hydroxide in 1000 cc. of solution) with an equal volume of water. To this solution add 4 or 5 drops of the ordinary concentrated hydrochloric acid. Stir the resulting solution with a glass rod and test its action on blue and on red litmus paper. (7) Has it acid or basic properties?

Now continue to add the acid drop by drop until the resulting solution is neutral (that is, has no effect on either blue or red litmus paper)

or is, at most, slightly acid. (8) Write the equation for the reaction between the hydroxide and the acid. Now pour the solution into an evaporating dish and evaporate to dryness. (9) What compound remains? (Taste it.)

(10) What is the name given to the compounds formed by the action of acids on bases? (11) Characterize acids and bases as to composition; as to their action on litmus; as to taste; as to their interaction with each other.

## EXERCISE 37

## THE RATIO OF ACID TO BASE IN NEUTRALIZATION

**Object.** To determine whether or not the reaction between acid and bases is a definite one.

**Apparatus.** 2 burettes and supports, as shown in Fig. 63; 2 small beakers and stirring rod; graduated pipette; small funnel.

**Materials.** Sodium hydroxide solution prepared by adding 20 cc. of the laboratory reagent to 80 cc. of water (this will contain approximately 2.4 g. of the hydroxide in 100 cc. of solution); 1 cc. of sulfuric acid (use graduated pipette) added to 100 cc. of water and mixed thoroughly; a few drops of a phenolphthalein solution (R.S.). (Phenolphthalein is a complex, nearly colorless compound whose aqueous solution turns a pink color when brought in contact with a base such as sodium hydroxide.)

*a.* (Two students may work together.) Rinse out a burette, first with distilled water and then with a little of the solution of sodium hydroxide. Support the burette (Fig. 63), place a funnel in the top of it, and pour the hydroxide solution into the tube until the level of the liquid is 1 or 2 cm. above the zero mark. Turn the stopcock and let the solution slowly flow out until the bottom of the meniscus (Fig. 2) of the liquid in the burette is on a level with the zero mark. In a similar way fill a second burette with the acid solution.

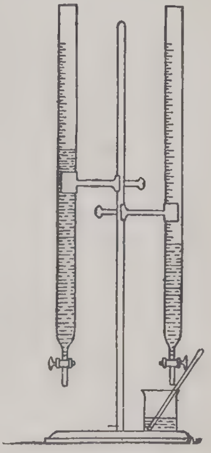


FIG. 63

Now let exactly 15 cc. of the acid solution flow into a small beaker, add 2 drops of phenolphthalein solution, and run in 2 or 3 cc. of the hydroxide solution. Notice that where the liquids come in contact a pink color is produced, which disappears quickly on stirring. Run in more of the solution, a little at a time, until the color fades slowly, and then a drop at a time until the entire liquid, on stirring, remains colored *faintly* pink. This marks approximately the point of neutralization. Record the number of cubic centimeters of the hydroxide solution used in the table. Repeat the experiment, using

25 cc. of the acid, and again record in the table the number of cubic centimeters of the hydroxide solution used to neutralize the acid and make the calculations called for.

### TABLE OF RESULTS

#### *First Experiment*

1. Volume of hydroxide solution used to neutralize 15 cc. of the acid . . . . . . . . . . . . . . . . . . . . . . cc.
2. Volume of hydroxide solution required to neutralize 1 cc. of acid (Calculate) . . . . . . . . . . . . . . . . . cc.

#### *Second Experiment*

3. Volume of hydroxide solution used to neutralize 25 cc. of the acid . . . . . . . . . . . . . . . . . . . . . . cc.
4. Volume of hydroxide solution required to neutralize 1 cc. of acid (Calculate) . . . . . . . . . . . . . . . . . cc.

(1) What do the results of the experiments prove?

Suppose that the sodium hydroxide solution used contained exactly 2.4 g. of NaOH in 100 cc. of solution. (2) Calculate from the results of the above experiments the number of grams of $H_2SO_4$ in 100 cc. of the sulfuric acid solution used.

The process of determining the strength (or concentration) of an acid by neutralizing it with a solution of a base of known strength is called *titration*. It is described by the chemist by stating that the acid is *titrated* with a base. Similarly, a base may be *titrated* with an acid.

**b.** (3) Distinguish between a *standard solution* and a *normal solution* (see text, page 273). Just as it is sometimes more convenient to use a solution one half the strength of the normal (half-normal) or one tenth the strength (tenth-normal), so it is sometimes better to use a solution twice as strong as the normal (2-normal) or five times as strong (5-normal). (4) Calculate the weight of HCl present in 1 liter of each of the following solutions of hydrochloric acid: normal; half-normal; 3-normal. (5) What weight of $H_2SO_4$ is present in 500 cc. of a tenth-normal solution of sulfuric acid? (6) Compute the weights of NaOH and KOH, respectively, that would be required to prepare 1 liter of a normal solution of each. (7) What volume of a normal solution of any given acid, such as hydrochloric acid, would be required to neutralize the base in 100 cc. of a 2-normal solution of a base such as sodium hydroxide?

## EXERCISE 38

## THE CONDUCTIVITY OF ACIDS, BASES, AND SALTS

**Object.** To determine whether or not certain substances conduct the electric current; also to determine the relative conductivities of certain acids, bases, and salts.

**Apparatus.** Current from an electric-lighting system; small beaker; porcelain crucible; apparatus as represented in Fig. 64 (*A* is an insulated wire with a plug on the end which will screw into the socket of a lighting system; *B* is an ordinary incandescent lamp; *C* is a metal spring so arranged that the current is broken unless the spring is pressed down; *D*, *D′* are rather stiff copper wires. The parts of the apparatus are connected by wires so that, when the spring *C* is pressed down, the current will flow from the wire *A*, through the spring *C*, through the wire *D*, through the liquid in the beaker, through the wire *D′*, through the lamp *B*, back to the wire *A*).

**Materials.** 5 g. of sodium chloride; 5 g. of sugar; sodium hydroxide solution; calcium hydroxide solution (limewater); sulfuric acid; hydrochloric acid; 10 cc. of a benzene solution of hydrogen chloride prepared by passing a current of *dry* HCl (made dry by causing the gas to bubble through sulfuric acid) into benzene (*remember that benzene is very inflammable*); 25 cc. of approximately 3-normal solutions of each of the following acids: hydrochloric, sulfuric, nitric, acetic; also approximately tenth-normal solutions of each of the above acids, prepared by diluting 3 cc. of the 3-normal solutions to 100 cc.; 0.04 g. of the indicator known as thymol blue dissolved in 100 cc. of water; 20 cc. of approximately 3-normal solutions of potassium hydroxide and sodium hydroxide; 20 cc. each of solutions containing 1 molar weight to the liter of sodium chloride, sodium nitrate, and sodium acetate, and one half molar weight of sodium sulfate; 1 g. of mossy zinc; 10 g. of potassium nitrate.

*a.* Obtain from your instructor the apparatus shown in Fig. 64. With emery paper polish the lower parts of the two wires *D*, *D′* until they are bright and free from any oxide. Attach the wire *A* to any convenient lamp socket. See that the wires *D*, *D′* are held firmly in place by the screws shown in the figure; then proceed to test the conductivity of the substances as described below.

*b.* Partly fill the small beaker with distilled water and hold it so that the ends of the wires dip into the water as shown in the figure. Now press down on the spring *C*, and note whether or not the water conducts the electric current. This is indicated by the glow of the lamp; moreover, the brightness of the glow will indicate roughly the relative conductivity of the water, or of whatever substance is in the beaker. (1) Does distilled water conduct the electric current?

*c.* Clean and dry the wires *D*, *D′*; then test the conductivity of dry, powdered sodium chloride (common salt) by placing some of it in a dry beaker, and holding the beaker so that the ends of the wires dip into the powder. (2) What is the result?

*d.* (3) Repeat *b*, using a solution of salt in distilled water.

*e.* (4) Test the conductivity of the following substances and interpret the results: dry powdered sugar; a solution of sugar in water; tap or well water; distilled water containing a few drops of sulfuric acid; distilled water containing a few drops of hydrochloric acid; a solution of sodium hydroxide; a solution of calcium hydroxide. (5) How do we account for the fact that solutions of some compounds conduct the electric current while others do not?

*f.* Obtain from your instructor 10 cc. of a benzene solution of hydrogen chloride (*benzene is inflammable*). (6) Test its conductivity. (7) Test its effect on blue and on red litmus paper. (8) Does it dissolve zinc? ✓ (9) How do you account for the difference in properties between a benzene solution of hydrogen chloride and an aqueous solution of the same gas?

*g. The relative strengths of acids determined by the relative conductivities of the acids.* (Four students may work together, each one being supplied with the conductivity apparatus shown in Fig. 64.) Compare the relative conductivities of the following acids: hydrochloric, sulfuric, nitric, and acetic. In making this comparison the normal solution of the acid is not concentrated enough to give the best results; hence use 3-normal solutions. Now partly fill the beaker in one apparatus with the standard solution of hydrochloric acid; similarly, partly fill the beaker in each of the other three sets with sulfuric, nitric, and acetic acids respectively. Connect the current and test the relative strength of these acids by comparing the glow of the light in each of the four sets of apparatus. (10) Designate the comparative strengths of the acid under the general headings "Strong," "Medium," "Weak."

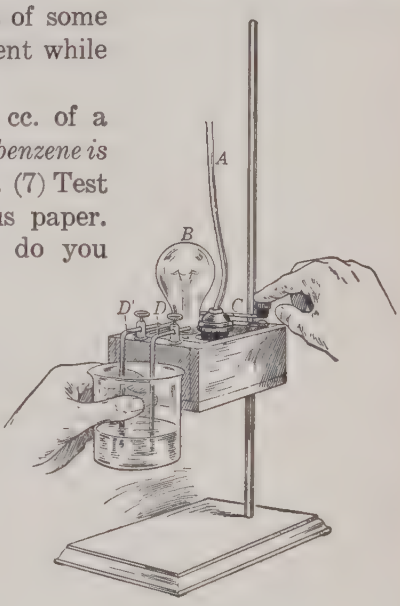


FIG. 64

*h. The relative strengths of acids determined by the action of the acids on indicators.* Pour 10 cc. of a tenth-normal solution of each of the above acids into separate test tubes. Now add to each 5 drops of the solution of thymol blue. Mix well and carefully note the intensity of the color change produced by each acid. The relative changes in color will be a measure of the relative strengths of the acids. (11) How do your results compare with those obtained in *g* above?

nitric
sulfuric

hydrochloric

cetic

Results are same, stronger acids make deeper color.

*i. Comparative degrees of ionization of bases; strength of bases.* (Use two sets of conductivity apparatus.) Proceed as in *g* to determine the relative strength of sodium hydroxide and potassium hydroxide, using 3-normal solutions. (12) Record your conclusions.

*j. Comparative degrees of ionization of salts.* Proceed as in *g* above, using solutions of the following salts prepared as directed under "Materials": sodium chloride, sodium nitrate, sodium acetate, sodium sulfate. (13) Record the results. (14) Do the salts of the acids show the same relative ionization as the corresponding acids?

*k. Conductivity of fused salts.* Fill a porcelain crucible about three fourths full of potassium nitrate. Heat the crucible and its contents until the nitrate is in a molten condition. Now immerse the ends of the two copper wires of the apparatus (Fig. 64) in the molten nitrate. Press down upon the spring *C* and note whether the electric current flows through the molten nitrate. The experiment may be repeated, using any salt that has a low melting point such as sodium nitrate, potassium acetate, sodium acetate, sodium nitrite. (15) Do the molten salts contain ions?

## EXERCISE 39

## DEGREE OF IONIZATION OF ELECTROLYTES

**Object.** To obtain some evidence for the dissociation of electrolytes when dissolved in water, and to calculate the extent of this in the case of a typical electrolyte.

**Apparatus.** The same as for Exercise 17.

**Materials.** Beaker of cracked ice, pieces the size of acorns; 100 g. of sodium chloride.

*a. Discussion.* Electrolytes lower the freezing point of water to a greater extent than do equimolecular quantities of nonelectrolytes. In addition, the lowering caused by a particular electrolyte is not necessarily the same as that shown by another. This abnormality of electrolytes is explained by assuming that an ion has the same effect as a molecule on certain physical constants of water (for example, the freezing point) as well as of certain other liquids. The extent of this abnormality is used as a measure of the degree of ionization of an electrolyte. Sodium chloride is a typical electrolyte and will be used in the following experiment.

The experiment is performed in exactly the same way as the one given under II in Exercise 17, except that sodium chloride is used in place of sucrose.

*b.* Determine the zero point on your thermometer, as explained in Exercise 17, II *a*; then proceed with the experiment exactly as described in Exercise 17, II *a* and *b*, except that about 15 g. (weighed to 0.01 g.) of sodium chloride is substituted for the sucrose. Insert the values in the following table:

### Table of Results

| | |
|---|---|
| 1. Freezing point of the water . . . . . . . . . . . . . . | ° C. |
| 2. Freezing point of the solution . . . . . . . . . . . . | ° C. |
| 3. Lowering of freezing point of the water . . . . . . . . | ° C. |
| 4. Weight of sodium chloride . . . . . . . . . . . . . . | g. |
| 5. Volume of solution . . . . . . . . . . . . . . . . . . | cc. |

*c. Calculations.* (1) From the above figures calculate the number of grams of sodium chloride in 1000 cc. of the above solution. What is the molar strength of this solution?

(2) Compare your results with the lowering of the freezing point of water produced by a molar weight of sucrose (see Exercise 17). Explain in detail how one accounts for this difference. Read the text on this point with great care (page 281).

(3) Calculate the molecular weight of sodium chloride on the assumption that no ionization takes place.

(4) From the results of this experiment calculate the degree of ionization of sodium chloride in the solution obtained.

## EXERCISE 40

## ELECTROCHEMICAL SERIES (DISPLACEMENT SERIES)

**Object.** To study the action of one metal on solutions of the salts of other metals.

**Apparatus.** 8 test tubes; test-tube rack.

**Materials.** 4 clean, bright strips each of zinc and copper (1 cm. × 10 cm.); 0.5 g. of lead nitrate dissolved in 10 cc. of water; 0.5 g. of copper nitrate dissolved in 10 cc. of water; 0.5 g. of mercuric nitrate dissolved in 10 cc. of water; 3 cc. of sulfuric acid dissolved in 10 cc. of water.

*a.* Pour into separate test tubes, to a depth of 4 or 5 cm., solutions of the following compounds: lead nitrate, dilute sulfuric acid, copper nitrate, mercuric nitrate. Set the tubes in a rack in the order given above and label them A, B, C, and D respectively.

Now place in each tube a strip of zinc. (It is convenient to have a strong thread attached to the upper part of each strip so that the strip may easily be withdrawn from the tube. The strips should be only partly immersed in the solution.)

After twenty minutes (in the meantime proceed with *b*) withdraw the strips and wipe them on a piece of white paper. (1) Note any evidence tending to show that the zinc has displaced the lead, hydrogen, copper, and mercury from their compounds. (As a rule, metals in a very finely divided form are black.) (2) Account for any change in the color of the copper nitrate solution.

*b.* Repeat experiment *a*, substituting for the zinc a strip of copper. (3) Contrast the results obtained with those obtained in *a*. (4) How

$15 : 54 :: x : 1000$

$54x \cdot 15000$

do you account for the change in the color of the solution of mercuric nitrate after the addition of the copper strip? (5) Are your results in accord with the table of the electrochemical series given on page 298 of the text?

## EXERCISE 41

## AMMONIA

**Object.** To prepare ammonia and to study its properties.

**Apparatus.** Test tube; hard-glass test tube fitted with cork and tubing as shown in Fig. 65; ring stand and burner; large beaker; three 250-cc. wide-mouthed bottles; 2 glass plates to cover bottles; piece of cardboard large enough to cover the mouth of a wide-mouthed bottle; glass rod.

**Materials.** Sodium hydroxide; 9 g. of ammonium chloride; 15 g. of powdered slaked lime ($Ca(OH)_2$); red litmus paper; blue litmus paper; 5 cc. of red litmus solution (R.S.); hydrochloric acid; wooden splint.

*a.* Dissolve 0.5 g. of ammonium chloride in 3 or 4 cc. of water in a test tube and heat to boiling. (1) Note the odor.

Now add 5 cc. of a solution of sodium hydroxide to the hot solution of ammonium chloride and continue the heating. (2) Again (*Caution*) note the odor. Moisten a strip of red litmus paper and hold it at the mouth of the tube but not in contact with it, and (3) note the results.

Dip the end of a glass rod in a concentrated solution of hydrochloric acid and hold it in the mouth of the test tube. (4) Account for the results.

(5) Complete the following equations:

$$NH_4Cl + NaOH \longrightarrow$$
$$NH_3 + H_2O \longrightarrow$$
$$NH_4OH + HCl \longrightarrow$$

*b. Usual laboratory method for preparing ammonia.* This differs from the method used in *a* only in the fact that the less expensive calcium hydroxide (slaked lime) is substituted for the sodium hydroxide. The form of apparatus used is shown in Fig. 65. The bottle *B* (250-cc.) contains 25 cc. of water. The glass tube *C* extends through a hole in a cardboard resting on the mouth of the bottle. The end of the tube must *just touch* the water in the bottle.

In the tube *A* place a mixture of 15 g. of powdered slaked lime and 8 g. of ammonium chloride. Connect the tube as shown in Fig. 65 and heat the mixture gently, beginning with that portion near the mouth of the tube and gradually extending the heat to the other portions. As soon as the gas is evolved freely (as shown by the bubbles at the end of the tube *C*), bring the tube *C* to an upright position, as shown in the dotted lines, and collect two bottles of the gas by displacement of air. To do this, bring each bottle successively down over the exit tube and hold it in this position until a drop of hydrochloric acid on the end of a glass rod fumes strongly when held at the mouth of the bottle; then withdraw the bottle, cover its mouth with a glass plate, and set it aside, mouth downward. (6) Why mouth downward?

When both bottles are filled, bring the tube *C* into the bottle *B* again and continue to heat the mixture gently as long as any gas is generated.

(7) Write the equations for all the reactions involved in the preparation.

*c. The properties of ammonia.* (8) Note the color and odor (***Caution***) of the gas. Is it heavier or lighter than air?

*d.* (9) Determine whether or not the gas is inflammable by bringing a lighted splint in contact with it. Describe the results.

*e.* Fill a large beaker with water and color it with a few drops of red litmus solution. Uncover the remaining bottle of the gas and at once bring its mouth under the surface of the water in the beaker. Leave it in this position for five minutes, taking care to keep the mouth of the bottle below the surface of the water. (10) What do the results prove?

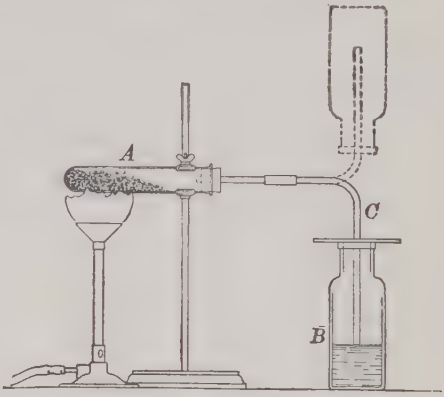


FIG. 65

*f.* (11) Note the odor of the liquid in the bottle *B*. (12) Try the effect of the liquid on blue and on red litmus paper. (13) How does it compare with the aqua ammonia of the druggist in its odor and its action on litmus? (14) Does the gas combine with the water, or is it simply dissolved in it? (15) Give reasons for your answer. Now neutralize the liquid with hydrochloric acid and evaporate just to dryness. (16) Compare the residue with the ammonium chloride used in experiment ***a***.

## EXERCISE 42

### AMMONIUM HYDROXIDE (AQUA AMMONIA)

**Object.** To determine the strength of the aqua ammonia sold by druggists and grocers.

**Apparatus.** Small glass bottle (the glass phials used by druggists serve well) and cork to fit; burette; graduated pipette; 250-cc. flask; stirring rod.

**Materials.** Normal solution of hydrochloric acid (or sulfuric acid) (R.S.); 1 or 2 cc. of a solution of methyl orange (R.S.) to use as an indicator; samples of the aqua ammonia obtained from the druggist or the grocer.

NOTE. The law requires that the aqua ammonia of the druggist and grocer must contain not less than 19.5 per cent by weight of $NH_4OH$, which corresponds to 9.5 per cent of $NH_3$. The strength of such solutions may be determined readily by titration with an acid of known strength as explained below.

Fill a burette with the normal solution of hydrochloric acid (any other convenient known strength may be used) as in Exercise 37. Weigh to 0.01 g. the glass phial and cork, recording the weight in the table below. Introduce into the phial about 5 cc. of the aqua ammonia to be tested, cork, again weigh, and record the weight. Pour about 50 cc. of water into the flask, and to this add the weighed sample of aqua ammonia, rinsing the phial and cork thoroughly with water, so that all the sample will be transferred to the flask. Use sufficient water so that there will be about 75 cc. of solution in the flask. Next add to this a sufficient amount of a solution of methyl orange to impart a distinct color to the liquid. Now from the burette (refer to Exercise 37) add the normal solution of hydrochloric acid until the orange color is just changed to a pink. The acid must be added slowly, a few drops at a time, and the mixture stirred thoroughly. As the point of neutralization is nearly reached, great care must be taken to avoid the addition of an excess of acid. Note and record the volume of acid used. Make the calculations indicated in the following table:

TABLE OF RESULTS

1. Weight of glass phial and cork . . . . . . . . . . . . . g. 47.11
2. Weight of glass phial and cork and aqua ammonia . . . . . g. 51.23
3. Weight of aqua ammonia in phial (Calculate) . . . . . . . g. 6.12
4. Weight of HCl in 1 liter of a normal solution (Calculate) . . g. 36.46
5. Weight of HCl in 1 cc. of a normal solution (Calculate) . . . g. .03646
6. Number of cubic centimeters of solution of acid used to neutralize the aqua ammonia . . . . . . . . . . . . . . . . 37.6
7. Weight of HCl in the volume of acid used . . . . . . . . . g.
8. Weight of $NH_4OH$ in the weight of aqua ammonia taken (Calculate from the weight of HCl required to neutralize it) . . g.
9. Percentage of $NH_4OH$ in the sample of aqua ammonia (Calculate) . . . . . . . . . . . . . . . . . . . . . %

Is the sample up to the requirements of the law?

## EXERCISE 43

## NITRIC ACID

**Object.** To prepare and to study the properties of nitric acid.

**Apparatus.** Glass retort (150-cc.), test tube, and beaker (500-cc.), arranged as shown in Fig. 66; funnel; evaporating dish; ring stand and burner.

**Materials.** 12 g. of sodium nitrate; 10 cc. of sulfuric acid; small piece of tin and of zinc; small strip of copper; pieces of ice the size of walnuts.

*a.* Arrange an apparatus like that shown in Fig. 66. Put into the retort *A* about 12 g. of sodium nitrate and 10 cc. of sulfuric acid, pouring the latter through a funnel placed in the tubulus *B* of the retort. Heat the mixture gently with a small flame. Nitric acid is set free, distills over, and is condensed in the test tube *C*, which is kept cold by being partly immersed in ice water in the beaker *D*. (1) Write the equation for the reaction involved.

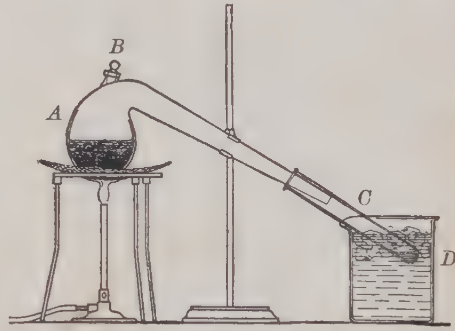


FIG. 66

*b.* When nitric acid is heated, a part of it is decomposed into water, nitrogen dioxide, and oxygen (the nitrogen dioxide is a reddish-brown gas which remains dissolved in the nitric acid, imparting to the acid a reddish-brown color). On this account it is a good oxidizing agent. To test its oxidizing properties, put a small piece of tin into a test tube, cover it with a little nitric acid, and gently heat (*Hood*). The white residue formed is composed mainly of a compound of tin and oxygen, the latter being supplied by the nitric acid.

*c.* Place a piece of zinc in a test tube and add a few drops of the acid. (2) Is a gas evolved? (3) If so, is the gas hydrogen (test with a burning splint)? (4) Account for the fact that the action of nitric acid on zinc differs from that of sulfuric and hydrochloric acids.

*d.* Place a small strip of copper in an evaporating dish (*Hood*) and add some of the acid you have prepared, a few drops at a time, until the copper is just dissolved. Evaporate the solution *just to dryness* (Fig. 59). (5) Note the appearance of the residue. Since copper is below hydrogen in the electrochemical series, how do you account for the fact that nitric acid dissolves the metal (study the reaction on page 312 of text)? Set the dish and residue ((6) What is the residue?) aside for use in Exercise 44.

## EXERCISE 44

## THE NITRATES

**Object.** To study the properties of the salts of nitric acid (nitrates).

**Apparatus.** Evaporating dish containing the copper nitrate prepared in *d*, Exercise 43; 5 test tubes in test-tube rack.

**Materials.** Strip of copper; crystal of lead nitrate; crystals of such nitrates as are available; sulfuric acid; 2 g. of ferrous sulfate dissolved in 10 cc. of water.

*a.* Heat the dish (*Hood*) containing the copper nitrate, prepared in Exercise 43, with a small flame. (1) Note the color of the gas evolved, also the color of the residue. (2) Compare the residue with a sample of copper oxide.

*b.* Place a crystal of lead nitrate in the evaporating dish and heat gently. (3) Compare with the results obtained in *a*.

*c.* Place a small crystal of each of such nitrates as are available in your laboratory in separate test tubes and test their solubility in water. (4) What nitrates are insoluble in water (page 313 of text)?

*d. How to detect the presence of nitrates.* Dissolve a crystal of sodium nitrate in 2 or 3 cc. of water in a test tube, *carefully* add, a few drops at a time, an equal volume of sulfuric acid, mixing the liquids after each addition and cooling with cold water to room temperature or below. The sulfuric acid acts on the nitrate, liberating nitric acid. Now slightly tip the tube and gently pour 2 or 3 cc. of the solution of ferrous sulfate down the side of the tube, so that it floats on the heavier liquid, and set the tube aside, being careful not to mix the two liquids. A brown ring soon forms where the liquids meet. Repeat the experiment, using potassium nitrate. This is a good test for nitrates. The brown ring is due to the presence of a complex compound formed by the action of ferrous sulfate on nitric acid.

## EXERCISE 45

## THE OXIDES OF NITROGEN

**Object.** To prepare the important oxides of nitrogen and to study their properties.

**Apparatus.** Hard-glass test tube, with delivery tube, as used in preparing oxygen (Fig. 29); 3 wide-mouthed bottles (250-cc.) and glass cover plates; pneumatic trough; hydrogen generator (Fig. 34).

**Materials.** 8 g. of ammonium nitrate; wooden splints; 5 small strips of copper; 10 cc. of nitric acid; 2 g. of potassium chlorate and 0.5 g. of manganese for the preparation of oxygen.

*a. Nitrous oxide.* Put 6 or 8 g. of ammonium nitrate in the hard-glass test tube used in the preparation of oxygen. Attach a delivery tube and heat *gently,* so as to cause a slow evolution of the gas.

As soon as the gas is regularly evolved, collect two or three bottles of it over water. Notice the water deposited on the sides of the test tube. (1) What is its source? (2) Note the color, odor, and taste of the gas. Test it with a glowing splint. (3) Account for the result. (4) How can you distinguish between nitrous oxide and oxygen?

*b. Nitric oxide and nitrogen dioxide.* Place a few pieces of copper in your hydrogen generator (***Hood***), just cover them with water, and add 2 or 3 cc. of nitric acid. After the air has been expelled from the generator fill two bottles (add more acid if necessary) with the evolved gas as in the preparation of hydrogen, and fill the third bottle half full, marking the volume of the gas by a rubber band slipped over the bottle.

Remove the two filled bottles of the gas, using glass plates to keep them covered, and leave the third bottle inverted in the water. Now uncover one of the bottles and (5) note and explain the result. (6) Test the gas in the second bottle with a burning splint.

Prepare some oxygen and, after the air is expelled from the apparatus, fill a bottle half full of the pure oxygen. Bring this alongside the bottle that is half filled with nitric oxide, and, keeping the mouths of the bottles under water, slowly invert the bottle of oxygen so that the oxygen is gradually transferred to the bottle containing the nitric oxide. (7) Note the change in color of the gas, also the change in volume after the gas has remained in contact with the water for about ten minutes. (8) Write the equation for the reaction between the nitric oxide and the oxygen. (9) In what proportion by volume do these two gases react (solve by Avogadro's principle)? The nitrogen dioxide formed reacts with the cold water to form a mixture of nitrous acid and nitric acid, which are gradually absorbed by the water. (10) Test any gas left in

the bottle, to find out whether it is nitric oxide or oxygen. (11) Are the results, including change in color and in volume of the gas, in general accord with the equation in question 8?

## EXERCISE 46

### EQUILIBRIUM

**Object.** To study reversible reactions.

**Apparatus.** 6 test tubes; test-tube rack; beaker; stirring rod.

**Materials.** Hydrochloric acid; barium chloride (R.S.); ammonium molybdate solution (R.S.); silver nitrate solution (R.S.); 0.1 g. of disodium phosphate; 0.5 g. of bismuth chloride; 1 g. of cupric bromide; 3 g. of potassium bromide; solution of ammonium thiocyanate (R.S.); solution of ferric chloride (R.S.); 3 g. of ammonium chloride; 1 g. of sodium chloride dissolved in 10 cc. of water; 0.3 g. of each of the following salts dissolved in separate portions (3 cc.) of water: potassium nitrate, common salt, borax, sodium carbonate, alum, cupric sulfate, sodium sulfate; blue and red litmus papers.

*a. Velocity of reactions.* To a dilute solution of sodium chloride add 1 or 2 cc. of a solution of silver nitrate. (1) How rapidly does the reaction take place? (2) How does it compare in rapidity with the reaction taking place when a little barium chloride is added to a solution of sodium sulfate? In general, reactions between freely ionized electrolytes are too rapid for measurement. Slow reactions occur when the reagents are very little ionized or when changes occur other than interchange between ions.

Dissolve a crystal of disodium phosphate no larger than a grain of wheat in 10 cc. of water and add 5 cc. of ammonium molybdate solution. (3) Does a precipitate at once form? (4) Do you notice any change of color? (5) Does this remain constant or increase? Set the tube aside and look at it from time to time during the laboratory period. (6) Is the formation of a precipitate gradual or sudden? (The yellow solid has a very complex formula.)

*b. Equilibrium and mass action.* Place a few crystals of bismuth chloride ($BiCl_3$) in a beaker and add about 1 cc. of water. (7) What change do you notice? The equation for the reaction is

$$BiCl_3 + H_2O \rightleftarrows BiOCl\downarrow + 2\,HCl$$

(8) Under what conditions should you expect the reaction to be reversed? Add concentrated hydrochloric acid, a drop at a time, with constant stirring. (9) Explain the results. When the solution is complete, add water, a little at a time, until the precipitate again appears. (10) Can you reverse the reaction again?

*c. Equilibrium in solution.* Dissolve about 1 g. of cupric bromide ($CuBr_2$) in the least possible volume of water. (11) What is the color of the solution? Add water, a few drops at a time, until the solution becomes a clear green. Pour off half the solution and dilute it further. (12) What is the final color? (13) How can you account for the intermediate green? To the remaining half of the green solution add crystals of potassium bromide or sodium bromide. (14) What change in color is noticed? (15) How do you explain this?

From the reagent shelf obtain about 5 cc. each of a solution of ammonium thiocyanate ($NH_4CNS$) and of ferric chloride ($FeCl_3$). Pour 1 cc. of each of these solutions into 50 cc. of water. An equilibrium results as follows:

$$3\,NH_4CNS + FeCl_3 \rightleftarrows Fe(CNS)_3 + 3\,NH_4Cl$$

The compound $Fe(CNS)_3$ (ferric thiocyanate) is deep red, and even in very dilute solution has a noticeable pink color. Half fill four test tubes with the solution and reserve one tube for comparison. To the second tube add 1 or 2 cc. of the solution of ammonium thiocyanate. (16) What change in color results? (17) Note the equation and explain. To the third tube add 1 or 2 cc. of the solution of ferric chloride. (18) Explain the change. To the fourth tube add 2 or 3 g. of solid ammonium chloride. (19) Explain the change.

*d. Hydrolysis.* Pour into separate test tubes small quantities of solutions of potassium nitrate, sodium chloride, borax (page 437 of text), sodium carbonate (page 535 of text), alum (page 599, 600 of text), and cupric sulfate (page 656 of text). Test the action of each on blue and on red litmus paper. (20) Note the results and explain.

## EXERCISE 47

## HYDROGEN FLUORIDE

**Object.** To prepare hydrogen fluoride and to study its properties.

**Apparatus.** Piece of window glass; small lead dish (laboratory outfit).

**Materials.** 2 or 3 small pieces of paraffin (size of a pea); 3 g. of calcium fluoride (fluorite); sulfuric acid.

***Caution.*** *Hydrogen fluoride is very corrosive and must not be inhaled; neither must its solution be brought in contact with the skin.*

Place some pieces of paraffin on a glass plate and gently warm the plate over a small flame. When the paraffin has melted, tilt the plate about so as to form a uniform layer of the wax. When the wax is cold, scratch your name *through* the wax with a pin. Place 3 g. of fluorite in a lead dish and add sufficient sulfuric acid to make a paste of it. Cover the dish tightly with the waxed side of the glass plate and set it in the hood for an hour; then scrape off the paraffin and examine the glass. (1) Describe the results and write the equations for all the reactions involved.

$CaCl_2$ + [illegible] $NH_3$ → [illegible]

$NH_4$ [illegible] + [illegible] $NO_3$ → [illegible]

$CaS_2$ + [illegible] $NH_4$ → [illegible]

## EXERCISE 48

## SOME FURTHER REACTIONS OF HYDROCHLORIC ACID AND ITS SALTS

**Object.** To study such reactions of hydrochloric acid and its salts as will enable one to detect their presence.

**Apparatus.** 6 test tubes.

**Materials.** Silver nitrate solution (R.S.); hydrochloric acid; nitric acid; ammonium hydroxide; crystals of different chlorides, such as those of sodium, calcium, and iron.

NOTE. Experiments on chlorine and hydrochloric acid were included under Exercises 33 and 34. The student should carefully review the results obtained, since they have an important bearing upon the experiments now to be performed with the remaining members of the chlorine family.

*a.* Add 4 or 5 drops of hydrochloric acid to 4 cc. of water, mix well, and add 2 or 3 drops of silver nitrate solution. (1) Note the results, and write the equation for the reaction.

Test the solubility of the precipitate in ammonium hydroxide and in nitric acid as follows: Shake the mixture in the tube and divide into two equal parts. To one add ammonium hydroxide until strongly alkaline; to the other add 3 or 4 drops of nitric acid. (2) Record your results.

*b.* Examine the physical properties of such chlorides as are available. Test their solubility in water. What ones are insoluble (page 263 of text)? Dissolve a small crystal of different chlorides each in 5 cc. of water and add silver nitrate solution as in *a.* (3) Write the equation for the reaction in each case. *The formation of a white precipitate (AgCl) with silver nitrate, which is soluble in ammonium hydroxide and insoluble in nitric acid, serves as a good test for hydrochloric acid and its salts.*

The silver nitrate ionizes in solution as follows:

$$AgNO_3 \rightleftarrows Ag^+ + NO_3^-$$

The hydrochloric acid and the chlorides ionize as follows:

$$\begin{aligned} HCl &\rightleftarrows H^+ + Cl^- \\ NaCl &\rightleftarrows Na^+ + Cl^- \\ CaCl_2 &\rightleftarrows Ca^{++} + Cl^-, Cl^- \end{aligned}$$

The silver ion $Ag^+$ and the chlorine ion $Cl^-$, when brought together in solution unite to form the insoluble silver chloride; hence the reaction goes to completion (page 336 of text). *The test with silver nitrate is therefore in reality a test for the chloride ion $Cl^-$.*

## EXERCISE 49

## BROMINE AND HYDROGEN BROMIDE

**Object.** To prepare bromine and hydrogen bromide and to study their properties.

**Apparatus.** Retort, test tube, and beaker, as shown in Fig. 67; burner; funnel; 2 test tubes; tripod; ring stand and clamp.

**Materials.** 3 g. of potassium bromide or sodium bromide; 4 g. of manganese dioxide; 10 cc. of sulfuric acid dissolved (*Caution*) in 40 cc. of water; strips of colored calico; 2 cc. of carbon tetrachloride; chlorine water (R.S.); sulfuric acid; blue litmus paper; 1 cc. of silver nitrate solution (R.S.).

***Caution.*** *The vapor of bromine must not be inhaled. Perform the experiments in a hood.*

*a. Preparation of bromine.* Put into the retort *A* (Fig. 67) a mixture of 2 g. of potassium bromide (or of sodium bromide) and 4 g. of manganese dioxide, and add to this through a funnel the cold dilute solution of sulfuric acid prepared as directed above. Shake the retort so as to mix the contents thoroughly. Note that the test-tube receiver *C* contains sufficient water to allow the end of the retort to dip just below its surface.

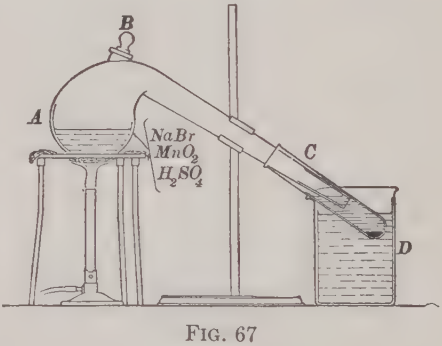


FIG. 67

Now heat the retort gently. The bromine is liberated and distills over. (1) Write the equation for the reaction. Continue the heating until all the bromine has distilled over. Remove the stopper from the retort before withdrawing the burner.

*b. The properties of bromine.* Study the properties of the bromine collected in the bottom of the test tube, (2) noting its color, odor, density, and solubility in water. (3) What property is implied in the name of the element? Add about 0.5 cc. of the aqueous bromine solution to 1 cc. of carbon tetrachloride. Note that the bromine solution does not mix with the carbon tetrachloride but floats on top of it. Now place your thumb over the mouth of the tube and shake the mixture vigorously for a few seconds; then set the tube aside until the two liquids separate. The bromine, being more soluble in carbon tetrachloride than in water, is taken up by the former liquid. (4) What is the color of the carbon tetrachloride solution?

(5) Test the bleaching property of bromine by immersing strips of colored cloth in the aqueous solution.

*c. Action of chlorine on sodium bromide.* Dissolve a bit of sodium bromide as large as a grain of wheat in 2 cc. of water. (6) What is the color of the solution? Now add to the solution 1 cc. of chlorine water. The chlorine displaces bromine from sodium bromide just as zinc displaces copper from a copper salt (page 298 of text). (7) Account for the change in the color of the solution.

*d.* (8) Summarize the properties and conduct of bromine.

*e. Preparation and properties of hydrogen bromide.* Add 3 or 4 drops of sulfuric acid to about 1 g. of potassium bromide in a test tube. Some hydrogen bromide is evolved, which attracts moisture as it escapes from the tube, forming a light cloud. The cloud may be made more noticeable by blowing across the mouth of the tube, thus furnishing additional moisture. (9) Test the vapor with a piece of blue litmus paper moistened with water. Along with the vapor of hydrogen bromide there appears in the tube a reddish vapor of bromine. There is also formed some sulfur dioxide (note carefully the odor to see if you can detect the sulfur dioxide). Recalling that hydrogen bromide is unstable, decomposing into hydrogen and bromine, and that concentrated sulfuric acid is an oxidizing agent (text, p. 360), (10) account for the results. (11) Distinguish between the terms *hydrogen bromide* and *hydrobromic acid.*

*f. Salts of hydrobromic acid (bromides).* Hydrobromic acid is unstable and is but little used. Its salts (bromides) are stable. Study the properties of potassium bromide as well as of any other available bromides. (12) Dissolve a crystal of potassium bromide in 5 cc. of water and apply the silver nitrate test as outlined for testing for chloride ions in Exercise 48. (13) Would this test alone serve to distinguish between chloride ions and bromide ions?

## EXERCISE 50

## IODINE AND HYDROGEN IODIDE

**Object.** To prepare and to study the properties of iodine and hydrogen iodide.

**Apparatus.** Beaker (250-cc.) and evaporating dish as shown in Fig. 68; 2 test tubes; test-tube rack; stirring rod; hydrogen sulfide generator; mortar and pestle.

**Materials.** 4 g. of sodium iodide or potassium iodide; 4 g. of powdered manganese dioxide; sulfuric acid; 2 cc. of alcohol; 2 cc. of carbon tetrachloride; 10 cc. of chlorine water (R.S.); 10 cc. of starch solution (R.S.); 0.5 g. of iodine; ferrous sulfide and hydrochloric acid for generating hydrogen sulfide; silver nitrate solution (R.S.); strip of filter paper dipped in a solution of lead acetate.

*a. Preparation of iodine.* Intimately mix on paper 3 g. of powdered sodium iodide and 2 g. of manganese dioxide. Place the mixture in the bottom of the beaker A (Fig. 68). Nearly fill the evaporating dish B with *cold* water. Now remove the evaporating dish, add 5 cc. of sulfuric acid to the mixture in the beaker, and stir it through the mixture; then at once place the evaporating dish back on the beaker as shown in the figure. Apply a very gentle heat to the mixture in the beaker, moving the burner about so as to heat the mixture uniformly. Note the colored vapor formed, which slowly condenses on the sides of the beaker and the bottom of the evaporating dish in the form of grayish-black crystals. Finally withdraw the flame and let the beaker stand until the vapor is nearly all condensed. (1) What is the substance? (2) Write the equation for its formation. (3) What property does the name of the substance suggest?

*b. Properties of iodine.* By means of a glass rod transfer a little of the iodine collected on the sides of the beaker in *a* to a test tube half filled with water, and shake it for one or two minutes. (4) Is the iodine soluble in water (note the color of the water) to any extent? Set the tube and contents aside for use below. Repeat, using alcohol in place of water. (5) Compare the solubility of the iodine in water and in alcohol. (6) What is the solution in alcohol called? Dissolve a small bit of the iodine in 2 cc. of carbon tetrachloride. (7) Note the color of the solution.

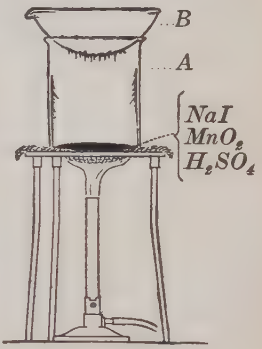


FIG. 68

*c. Action of chlorine on sodium iodide.* Dissolve a bit of sodium iodide as large as a grain of wheat in 2 cc. of water in a test tube. (8) What is the color of the solution? Now add to the solution 1 cc. of chlorine water. (9) Account for the change in the color of the solution.

*d. Action of iodine on starch.* Place two test tubes in your test-tube rack and label them No. 1 and No. 2. Half fill each with starch solution. To No. 1 add a few drops of the aqueous solution of iodine reserved in experiment *b*, above. To No. 2 add a bit of sodium iodide about as large as a grain of wheat and shake the mixture until the iodide dissolves. (10) Does free iodine change the color of starch solution? (11) Does iodine in combination change the color of starch solution? Now add 1 or 2 cc. of chlorine water to tube No. 2 and shake the mixture. (12) Note and account for the results. (If the color is very deep, pour out a portion of the liquid and replace it with water.)

*e. Hydrogen iodide and hydriodic acid.* Hydrogen iodide is still less stable than hydrogen bromide. What reaction would you expect to take place when concentrated sulfuric acid is added to a crystal of potassium iodide? (Compare *e*, Exercise 49.) Try the reaction and (13) note any evidences in favor of your opinion.

1. [illegible]

2. $2NaCl + 2H_2SO_4 + MnO_2 \rightarrow Na_2SO_4 + MnSO_4 + 2H_2O$
$+ Cl_2$

3. [illegible]

4. [illegible]

5. [illegible]

6. [illegible]

7. [illegible]

8. [illegible]

9. [illegible]

10. [illegible] standard solution

11.

Hydrogen iodide may be prepared by a number of methods, one of which is as follows: Grind to a fine powder 2 or 3 crystals of iodine in a mortar and add a little water. Transfer the resulting mixture to a test tube, adding sufficient water to nearly fill the tube. Now pass a slow current of hydrogen sulfide through the liquid until the iodine all disappears. Filter off the white solid (sulfur) and boil the filtrate until the hydrogen sulfide is expelled (test the vapor with lead acetate paper). Add a few drops of a solution of silver nitrate to the resulting liquid. (14) Note and account for the results.

(15) Distinguish between the terms *hydrogen iodide* and *hydriodic acid.*

*f. The salts of hydriodic acid; the iodides.* While hydriodic acid is unstable, its salts are stable. Study the properties of potassium iodide. Prepare a solution of the salt and study the action of silver nitrate upon it, as in the case of chlorides and bromides (Exercises 48 and 49). (16) Note the results, writing the equation for the reactions involved.

## EXERCISE 51

### SALTS OF THE BINARY ACIDS OF THE HALOGENS

**Object.** To find methods for distinguishing between chlorides, bromides, and iodides.

**Apparatus.** 3 test tubes; test-tube rack; mortar and pestle.

**Materials.** 1 g. each of sodium chloride, sodium bromide, sodium iodide; 1 cc. of silver nitrate solution (R.S.); nitric acid; 10 cc. of chlorine water (R.S.); 5 cc. of carbon tetrachloride (R.S.); 2 g. of manganese dioxide; sulfuric acid.

*a. Test for the ions of chlorides, bromides, and iodides* ($Cl^-$, $Br^-$, $I^-$). Place three test tubes in your rack and label them No. 1, No. 2, and No. 3, respectively. Put into No. 1 a bit of sodium chloride as large as a grain of wheat, in No. 2 a like amount of sodium bromide, and in No. 3 the same amount of sodium iodide. Now add to each tube about 2 cc. of water and shake the mixtures until the solids are dissolved; then add to each tube 2 or 3 drops of silver nitrate solution. (1) Describe the results. (2) Write the equation for the reaction in each case. Add to each tube 4 or 5 drops of nitric acid and shake the mixture. (3) Does the precipitate dissolve? The reaction with silver nitrate serves to detect the presence of the ions of chlorides, bromides, and iodides.

*b. To distinguish between chlorides, bromides, and iodides (method 1).* Arrange three test tubes as directed in experiment *a*, above. Thoroughly mix in a mortar a bit of sodium chloride as large as a pea with an equal amount of manganese dioxide and transfer the mixture to tube No. 1. Put in tube No. 2 a similar mixture of sodium bromide and manganese dioxide, and in tube No. 3 a like mixture of sodium iodide and manganese dioxide. Set the tubes in the hood and add to each 4 or 5 drops of sulfuric acid. If the reaction is not marked, heat the tubes gently. (4) How can you distinguish between chlorides, bromides, and iodides?

*c. To distinguish between chlorides, bromides, and iodides (method 2).* Again arrange three clean test tubes in your rack. Prepare in tube No. 1 a solution of sodium chloride as directed in experiment *a*, above; in a similar way prepare in tubes No. 2 and No. 3 a solution of sodium bromide and of sodium iodide respectively. Now add to each solution about 1 cc. of carbon tetrachloride (note that the tetrachloride does not mix with the water but sinks to the bottom of the tube). Shake the mixtures and then set them in the rack for a few seconds until the liquids separate. (5) Note the color of the carbon tetrachloride. Now add to each tube about 2 cc. of chlorine water and again shake the contents of the tubes vigorously; then set the tubes aside until the liquids again separate. (6) Now note the color of the carbon tetrachloride. (7) What is the function of the chlorine water? (8) Describe in detail the method for distinguishing between chlorides, bromides, and iodides by using chlorine water and carbon tetrachloride. Note that the silver nitrate test will enable you to tell whether or not a given compound belongs to the group composed of chlorides, bromides, and iodides. Either the test with manganese dioxide and sulfuric acid or the test with chlorine water and carbon tetrachloride will enable you to distinguish the three members of the group from each other.

***d.*** Obtain from your instructor an unknown sample of a chloride, bromide, or iodide, and determine its identity.

## EXERCISE 52

## POTASSIUM HYPOCHLORITE; POTASSIUM CHLORATE; BLEACHING POWDER

**Object.** To prepare potassium hypochlorite, potassium chlorate, and bleaching powder, and to study their properties.

**Apparatus.** A chlorine generator arranged according to Fig. 69 (the bottle *B* is about one third full of water); also the apparatus shown in Fig. 70; 4 test tubes (two of these should be good-sized); 4 beakers; glass cover for one of the beakers; carbon dioxide generator; funnel; 4 or 5 filter papers.

**Materials.** 6 g. of potassium hydroxide; 30 g. of manganese dioxide; hydrochloric acid; sodium hydroxide solution; nitric acid; silver nitrate solution (R.S.); 3 cc. sulfuric acid dissolved (***Caution***) in 10 cc. of water; strips of brightly colored calico; sulfuric acid; calcium hydroxide sufficient to half fill the tube *G* (Fig. 70); pieces of marble for generating carbon dioxide; splint.

*a. Hypochlorites and chlorates.* Place about 6 g. of potassium hydroxide in a small beaker, pour in 18 cc. of water, and stir and cool the liquid until the solid is all dissolved. Pour half the solution into a good-sized

test tube and label it No. 1. Pour the remaining half into a similar tube and label it No. 2. Cool the solution in tube No. 1 with cold water. Next generate (***Hood***) a slow current of chlorine in flask *A*, using about 15 g. of manganese dioxide and 75 cc. of hydrochloric acid, following directions given in Exercise 33. Arrange the exit tube *F, D, E,* as shown in Fig. 69 so that the chlorine generated will bubble slowly through the solution in tube *E* (No. 1). This tube is kept partly immersed in cold water, so as to keep the solution cold. Continue to pass the chlorine through the solution until no more chlorine is absorbed (this will ordinarily take from 10 to 15 minutes). Now withdraw the test tube from the exit tube and replace it by test tube No. 2, the contents of which have been previously heated until the tube is uncomfortably warm to the hand. This temperature should be maintained by a gentle heat while the chlorine is bubbling through the solution. Continue passing chlorine through the solution until it is no longer absorbed.

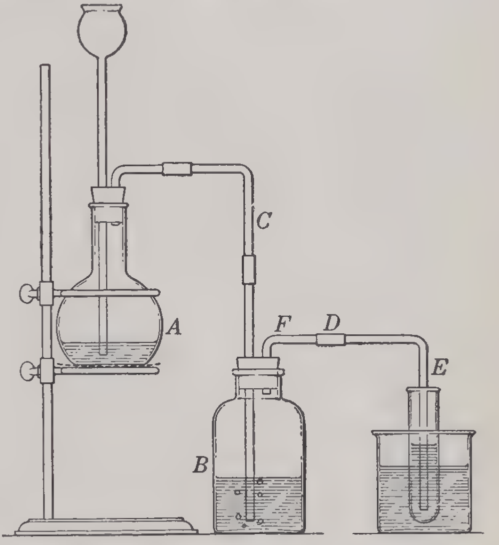


FIG. 69

Now pour the contents of tube No. 1 into a small beaker. Test 2 or 3 cc. of the solution for the chloride ion (Exercise 48). (1) Results. (2) Why is the chlorine passed through the water in bottle *B*? Immerse a strip of brightly colored calico in the remainder of the solution. (3) Is it bleached? Add to the solution 3 or 4 cc. of the dilute sulfuric acid. (4) Does the addition of the acid cause the cloth to be bleached more rapidly? (5) Explain.

Next pour the solution in tube No. 2 into a beaker, cover the beaker and set it aside until the next laboratory period. Filter off the crystals and wash them on the filter paper with cold water until the filtrate no longer gives the test for the chloride ion. (6) Account for the presence of the chlorides in the filtrate. Dry the remaining crystals by pressing them between filter papers; then transfer the solid to a test tube, apply a strong heat, and test for evolved oxygen. (7) Account for the results, writing the equations for all the reactions involved.

*b. Bleaching powder.* Disconnect the tube *F* at *D* (Fig. 69) and connect the generator with the tube *G* and bottle *H* (Fig. 70). The tube *G* is about one third filled with dry calcium hydroxide, while the bottle *H* is about one fourth filled with a solution of sodium hydroxide. Recharge the generator flask *A* with about 15 g. of manganese dioxide and add 75 cc. of hydrochloric acid. The chlorine must be evolved slowly, so that only a *very gentle heat* is applied to the flask. (8) What is the function of the sodium hydroxide in *H*? When the chlorine ceases to be evolved (note the color of gas in generator), disconnect the apparatus.

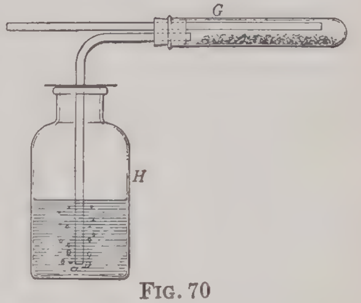


FIG. 70

(9) Write the equation for the reaction that takes place in the formation of bleaching powder.

Place about half of the bleaching powder prepared above, in a small beaker, and cover the beaker with a glass plate to the under side of which adheres a strip of moist, colored calico. Add to the beaker 10 or 15 cc. of a solution of sulfuric acid prepared by mixing (***Caution***) equal volumes of sulfuric acid and water. (10) Is the cloth bleached? (11) What is the function of the sulfuric acid?

Place the remainder of the bleaching powder in a small beaker and pour over it about 50 cc. of water. Stir the mixture for two or three minutes and filter. (12) What is the composition of the solid? (13) What passes into solution? Now pass into the filtrate a slow current of carbon dioxide as long as a precipitate continues to form; then filter. (14) What is the composition of the precipitate? (15) What compounds are present in the filtrate? Immerse strips of colored calico in the filtrate. (16) Record your results and explain.

(17) Show by equations the difference between the action of sulfuric acid and of carbonic acid on bleaching powder. (18) Aside from bleaching, what are the chief uses of bleaching powder?

## EXERCISE 53

### SULFUR

**Object.** To study the properties and chemical conduct of sulfur.

**Apparatus.** 3 test tubes; thermometer graduated to not less than 125°; smallest beaker; magnifying glass; porcelain crucible; pipestem triangle; large beaker; iron forceps; 15 cm. of glass tubing; wire gauze.

**Materials.** 5 cc. of carbon disulfide; small piece of brimstone; 20 g. of powdered brimstone; strip of copper foil (0.5 × 3 cm.); 5 g. of iron powder; 0.5 g. of cupromercuric iodide, or sealed tube containing this (see Appendix, D, 2).

***Caution.*** *Keep carbon disulfide away from flame and do not inhale its vapor.*

***a.*** *Properties of sulfur.* (1) Examine the physical properties of a piece of brimstone. Pour 2 or 3 cc. of carbon disulfide (***Hood***) over 2 g. of powdered brimstone in a test tube. Cover the mouth of the tube with

the thumb and shake the contents gently until the sulfur is dissolved, adding more carbon disulfide if necessary. Pour the clear solution into a small beaker, cover the beaker loosely with a filter paper, and set it aside in the hood. The carbon disulfide soon evaporates, the sulfur being deposited in crystals. (2) Examine these with a magnifying glass, noting their general shape. (3) What is this form of sulfur called (page 379 of text)? Save the crystals to compare with others obtained later in the exercise.

Half fill a test tube with powdered brimstone, dip the bulb of the thermometer into the powder, and heat the tube gently until the sulfur begins to melt; then continue the gentle heating, stirring the product with the thermometer until it is just melted. (4) Note the melting point of the sulfur. (5) Note the properties of the liquid. Now withdraw the thermometer and apply a stronger heat and observe that the liquid becomes darker, and at a certain temperature (about 235°) is so thick that the tube may be inverted without spilling it. Finally, increase the heat until the sulfur boils (444.6°), and then slowly pour the boiling liquid into a beaker of cold water. (6) Examine the product. (7) What name is given to this form of sulfur? Expose it to the air for an hour or longer. (8) Have its properties remained unchanged?

Fill a porcelain crucible with powdered brimstone and apply a very gentle heat until the sulfur is just melted. Withdraw the flame and examine the liquid carefully as it cools. Crystals soon begin to form on the surface of the melted sulfur, rapidly extending from the circumference toward the center. Before they reach the center, grasp the crucible with the forceps and quickly pour off the remaining liquid and examine the crystals. (9) Compare them in shape with those deposited from the solution of carbon disulfide. (10) In how many forms have you obtained sulfur?

*b. Chemical conduct of sulfur.* Burn a small piece of sulfur. (11) Notice the appearance of the burning sulfur and the odor of the gas ($SO_2$) formed.

Boil a little sulfur in a test tube and drop a small strip of copper foil heated to redness into the boiling liquid. (12) Is there any visible evidence of a chemical change? (13) What is formed?

Grind together 5 or 6 g. of iron powder with an equal weight of sulfur, transfer the mixture to a test tube, and heat it strongly in a Bunsen flame. (14) Describe the results. Retain the solid formed for use in the following exercise. (15) Summarize the properties and conduct of sulfur.

*c. Transition point.* The transition point for rhombic and monoclinic sulfur is at 95.5° (page 379 of text). Such a transition is always accompanied by changes in density, crystalline form, refractive index, change in solubility, and sometimes by marked color changes. For the most part the temperature of transition is not easy to measure, because the changes are not large and there is often a good deal of delay in the change. To obtain the exact temperature the measurements must extend over a considerable time. The change of rhombic sulfur into monoclinic sulfur is of this kind and its measurement is not suited to a short laboratory period. In some cases, however, the transition is very prompt, and with marked color change, and the transition temperature in such a case is easily determined. The following is a striking example.

Obtain a piece of thin-walled glass tubing about 15 cm. long and 5 mm. in diameter. Seal one end in the Bunsen flame, blowing the seal into rounded form like a test tube. Obtain about half a gram of cupromercuric iodide (R.S.) and put it into this test tube. Half fill a beaker with water, place it on a wire gauze on a ring stand, and suspend a thermometer in such a way that the bulb dips into the water. Heat the water until the temperature reaches about 60°, then turn the flame down so that the rise in temperature is very slow. Use the tube containing the scarlet powder as a stirring rod and watch for a change in color. (16) What change occurs? (17) At what temperature? Withdraw the flame, allowing the temperature to fall slowly. (18) At what point does the reverse change occur? Repeat the experiment to ascertain within what limits of temperature the change occurs. Return the powder to the stock bottle. Cupromercuric iodide is sometimes used as a paint for bearings in machinery, since its change in color indicates that the bearing is becoming heated.

NOTE. If desired, the student may himself prepare the cupromercuric iodide as follows: Dissolve 0.2 g. of mercuric chloride in 2 or 3 cc. of hot water in one test tube, 0.2 g. of potassium iodide in another, 0.3 g. of copper sulfate in a third, and 0.3 g. of sodium sulfite in a fourth. Add the potassium iodide, drop by drop, to the mercuric chloride until the bright scarlet precipitate just redissolves. To the solution of sodium sulfite add 2 cc. of dilute sulfuric acid (1 : 5), and pour this solution into the one just prepared; then quickly add the copper sulfate. Collect the precipitate of red cupromercuric iodide on a filter paper, wash it once or twice with cold water, and dry the powder on sheets of filter paper.

## EXERCISE 54

## HYDROGEN SULFIDE

**Object.** To prepare hydrogen sulfide and to study its properties.

**Apparatus.** Test tubes; hydrogen generator connected as shown in Fig. 35, except that the tube *B* is filled with cotton wool; glass tube and bottles, so that the hydrogen generator can also be used as shown in Fig. 71; 2 wide-mouthed bottles (250 cc.); bottle holding about 50 cc.; funnel and filter paper; conductivity apparatus (Fig. 64); small beaker.

**Materials.** The ferrous sulfide formed in *b*, Exercise 53; 10 g. of ferrous sulfide (pieces as big as a bean or larger); 20 cc. of hydrochloric acid diluted with an equal volume of water; blue and red litmus papers; silver coin; nitric acid; wooden splint.

*Caution. Hydrogen sulfide is poisonous if breathed in a form that is at all concentrated.*

*a. Preparation and properties of hydrogen sulfide.* Place a small piece of the ferrous sulfide formed in *b*, Exercise 53, in a test tube and cover it with the dilute hydrochloric acid. (1) Note the color and odor of the gas evolved. (2) Write the equation for the action of the hydrochloric acid on ferrous sulfide.

(*Hood.*) (*Usual laboratory method.*) Connect the hydrogen generator as shown in Fig. 35, filling the tube *B* with cotton wool. Put into the generator some pieces of ferrous sulfide, insert the stopper, and add water through the funnel tube until the end of the tube dips just below the surface of the water; then pour in 5 cc. of the dilute hydrochloric acid, adding more from time to time to maintain a gentle evolution of the gas. After the air has been expelled from the generator, ignite the gas at the end of the jet. Note the odor of sulfur dioxide. Hold a dry beaker over the flame to obtain evidence of the formation of water. (3) Write the equation for the combustion of hydrogen sulfide. Hold a beaker partly filled with cold water down in the flame of the burning gas for a few seconds. (4) Note and explain the results.

*b. Hydrosulfuric acid.* Remove the jet *C* from the tube *B* (Fig. 35) and connect the tube *B* with the bottle *C*, as shown in Fig. 71. Pour into this bottle a few cubic centimeters of water, so that the hydrogen sulfide generated must bubble through it. In this way the water will remove from the hydrogen sulfide any hydrochloric acid carried over with it. Now generate hydrogen sulfide in the apparatus, passing the washed gas into about 60 cc. of water in *D* until the latter is saturated with the gas. (5) What is this solution called? Place a few drops of the solution on strips of red and of blue litmus paper. (6) Note your results. Test the conductivity of the solution as in Exercise 38.

Taste a few drops of the solution. (7) What conclusions do you draw from these experiments as to the nature of the solution? Immerse a silver coin in the solution and (8) note and explain the results.

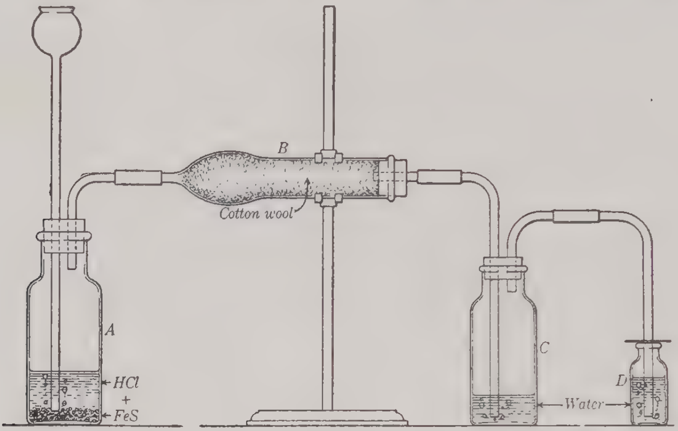


FIG. 71

Pass a few bubbles of hydrogen sulfide through 3 cc. of concentrated nitric acid. Sulfur separates as a white solid. (9) Account for the formation of the sulfur.

Filter off the liquid left in the generator *A*, collecting the filtrate in a small beaker. Set it aside in the hood until the next laboratory period. (10) What is the crystalline substance deposited?

*c.* (11) Summarize the properties and chemical conduct of hydrogen sulfide and of hydrosulfuric acid.

## EXERCISE 55

### SALTS OF HYDROSULFURIC ACID — SULFIDES

**Object.** To prepare the salts of hydrosulfuric acid and to study their properties.

**Apparatus.** Hydrogen generator and connections, as shown in Fig. 72; 6 test tubes; funnel and filter papers; watch glass.

**Materials.** Ferrous sulfide and dilute hydrochloric acid, as used in Exercise 54; separate solutions of silver nitrate, copper sulfate, cadmium chloride, lead nitrate, zinc sulfate, and sodium chloride, made by dissolving about 0.3 g. of the solid in 5 cc. of water (solutions on the reagent shelf may be used); sulfuric acid (2 cc. of the acid added to 5 cc. of water (*Caution*)); lead acetate (R.S.).

*a. Preparation of sulfides.* Arrange the hydrogen sulfide generator as shown in Fig. 72 and charge it as directed in Exercise 54 (***Hood***). The delivery tube *C* dips into the salt solution contained in the test tube *B*. Pass a few bubbles of the gas through each of the following solutions:

silver nitrate, copper sulfate, cadmium chloride, sodium chloride, lead nitrate. The exit tube *C* through which the gas bubbles into the solutions must be thoroughly cleaned each time. (1) Note the color of the precipitates obtained. (2) Write the equations for the reactions involved. (3) Do any of the solutions fail to give a precipitate when the gas is passed through them? (4) How do you account for this?

*b.* Pass the gas into a solution of zinc sulfate as long as a precipitate continues to form. (5) If the action is a completed one, will any zinc sulfate remain in the solution? Filter from the zinc sulfide and add a little ammonium hydroxide to the filtrate. (6) How do you account for the additional precipitate?

*c. Test for hydrogen sulfide.* Dip a strip of filter paper into a solution of lead acetate. Remove the cork from the hydrogen sulfide generator and insert the paper for a moment. (7) Note and account for the results. This serves as a convenient test for the gas. (8) What other property would serve to detect it if present in any marked quantity?

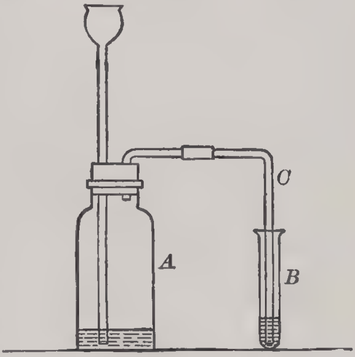


FIG. 72

*d. Test for sulfides.* Filter off the precipitated sulfides obtained in *a*, and wash them with water until the odor of hydrogen sulfide is no longer noticeable. Transfer each in succession to a watch glass and add to the solid 1 or 2 drops of dilute sulfuric acid. (9) Carefully note the odor. Most of the sulfides, when treated in this way, evolve hydrogen sulfide, which can be detected by its odor as well as by its action on paper moistened with lead acetate.

Heat a little ferrous sulfide in the flame of the burner. The sulfur in the sulfide burns, forming $SO_2$. (10) Note the odor. All sulfides, when heated in oxygen or air, form $SO_2$, just as the ferrous sulfide does.

*e.* (11) Summarize the three tests for sulfides.

## EXERCISE 56

## SULFUR DIOXIDE AND SULFUROUS ACID

**Object.** To prepare sulfur dioxide and sulfurous acid and to study their properties.

**Apparatus.** 250-cc. flask fitted with funnel tube and glass exit tube, as shown in Fig. 73; 3 bottles (250-cc.); watch glass; medicine dropper.

**Materials.** 10 g. of copper; sulfuric acid (2 cc. of the acid added to 5 cc. of water (*Caution*)); splint; blue litmus paper; crystal of sodium sulfite; strips of colored calico or a red flower.

*a.* (1) By what method have you already prepared sulfur dioxide (Exercises 6, 53, 54, 55)?

*b. Preparation of sulfur dioxide and sulfurous acid by the action of sulfuric acid on copper (laboratory method).* Place about 10 g. of copper turnings or small pieces of sheet copper in a generator arranged as in Fig. 73. Add 25 cc. of concentrated sulfuric acid and apply a gentle heat. As soon as the action begins, lower the flame, regulating it so as to obtain a uniform evolution of the gas. Collect two bottles of the gas by displacement of air; then cause it to bubble through 25 cc. of water as long as any is dissolved. (2) Write the equation for the reaction of sulfuric acid on copper (page 386 of text).

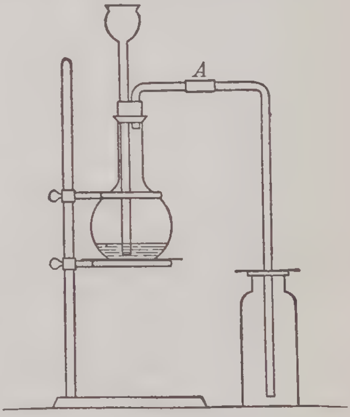


FIG. 73

*c. Properties and chemical conduct of sulfur dioxide and sulfurous acid.* (3) Note the odor of the gas. (4) Is the gas combustible? (5) Test with blue litmus paper the liquid formed by passing the gas into water. (6) Does the gas combine with the water or simply dissolve in it? (7) Give reasons for your answer. (8) Immerse in the liquid some small strips of colored calico or some petals of a red flower and see whether there are any results.

*d. Salts of sulfurous acid (sulfites).* Place a small crystal of some sulfite, such as sodium sulfite ($Na_2SO_3$), on a watch glass and moisten it with 2 or 3 drops of dilute sulfuric acid. (9) Note the odor of the evolved gas. *All sulfites evolve sulfur dioxide when treated with sulfuric acid.* This reaction serves as a good test for sulfites. It also serves as a method for preparing sulfur dioxide.

*e. Summary.* (10) Enumerate three methods for preparing sulfur dioxide. (11) Briefly summarize the properties and chemical conduct

of sulfur dioxide. (12) In what respect are carbon dioxide and sulfur dioxide alike? (13) In what respect are the sulfites and the carbonates alike? (14) Write equations to illustrate these similarities.

## EXERCISE 57

## SULFURIC ACID

**Object.** To study the properties of sulfuric acid.

**Apparatus.** 500-cc. flask fitted with a 2-holed stopper; glass tubing; apparatus used in generating sulfur dioxide (Fig. 73); test tubes.

**Materials.** 1 cc. of fuming nitric acid; strips of copper for generating sulfur dioxide; sulfuric acid; small piece of charcoal the size of a bean; piece of mossy zinc; 0.5 g. of sugar; barium chloride (R.S.).

*a. Preparation.* Fit a 500-cc. flask with a 2-holed stopper. Through one hole insert a right-angled glass tube so that the end will extend about halfway to the bottom of the flask. A short open glass tube extends just through the other hole of the cork. Warm the flask, pour into it 3 or 4 drops of the fuming nitric acid, and insert the cork and glass tubes. Gently move the flask so that the nitric acid will be vaporized by the warm sides of the flask. Now connect the longer glass tube with the sulfur dioxide generator and pass in a slow current of the gas until the gas in the flask is just decolorized. Allow the flask to stand and note the gradual formation of crystals on the sides of the flask. Now remove the cork and tubes from the flask, add a few drops of warm water, and shake the flask. Save the resulting liquid to test for the presence of sulfuric acid. (1) Note your results and account for the formation of sulfuric acid (pages 392, 393 of text).

*b. Properties and chemical conduct.* (2) Recall properties of sulfuric acid already studied, including its acid properties (Exercises 36, 38, and 40), its action on metals (Exercises 8 and 56), its action on salts (Exercises 34, 43, 55, and 56), and its action as a drying agent (Exercise 33).

Heat a bit of charcoal with 1 or 2 cc. of sulfuric acid. (3) What gas is evolved (odor)? (4) Account for the formation of this gas, recalling that carbon has a strong affinity for oxygen. (5) Place a piece of mossy zinc in the bottom of a test tube and add 2 or 3 cc. of concentrated sulfuric acid. Heat gently and (6) note the gas evolved (detect by odor). (7) Account for the difference in the action of dilute sulfuric acid and of concentrated sulfuric acid on metals.

Put a drop of concentrated sulfuric acid on a splint. Pour a few drops on 0.5 g. of sugar in a test tube. Examine after a few minutes. (8) Account for the results.

*c. Test for sulfuric acid.* Add 3 drops of sulfuric acid (*Caution*) to 5 cc. of water in a test tube. To this add a few drops of a solution of barium chloride. Note that a precipitate forms. Now add 3 or 4 drops of hydrochloric acid. Does the precipitate dissolve? *The formation with barium chloride of a precipitate which is insoluble in hydrochloric acid* constitutes a good test for sulfuric acid. (Sulfates also give this test, as will be explained in Exercise 58.) (9) Write the equation for the reaction. Test the liquid formed in *a* of this exercise for the presence of sulfuric acid, and (10) record the result.

## EXERCISE 58

### SALTS OF SULFURIC ACID—SULFATES

**Object.** To study the properties and preparation of sulfates.

**Apparatus.** 6 test tubes; evaporating dish; ring stand; burner; funnel; beakers; crucible; filter paper; litmus paper.

**Materials.** Crystals or small quantities (0.1 g.) of the sulfates available in the laboratory; 2 cc. of barium chloride solution (R.S.); hydrochloric acid; 7 g. copper oxide; 15 g. potassium hydroxide; sulfuric acid.

*a.* (1) Examine the physical properties of sodium sulfate, calcium sulfate, barium sulfate, copper sulfate, magnesium sulfate, ferrous sulfate, and such other sulfates as are available.

Test the solubility of each of the above sulfates in water. (2) What sulfates are insoluble (page 396 of text)?

*b. Test for sulfates.* Prepare a dilute solution of different sulfates by dissolving a crystal of each in 2 or 3 cc. of water. Add to each 1 drop of barium chloride solution. (3) Note and account for the result. Add 1 or 2 drops of hydrochloric acid to the mixture in each tube. (4) Does the precipitate dissolve? *A solution of barium chloride added to a solution of any sulfate gives a white precipitate which is insoluble in hydrochloric acid.* This reaction serves as a good test for sulfates.

It will be noted that with barium chloride both sulfuric acid and its salts give the same product, namely, a white precipitate of barium sulfate. This is evident from the following facts. It will be recalled that both acids and salts are ionized in solution. In the case of sulfuric acid and sulfates, ions are formed as follows:

$$H_2SO_4 \rightleftarrows H^+, H^+ + SO_4^{--}$$
$$Na_2SO_4 \rightleftarrows Na^+, Na^+ + SO_4^{--}$$
$$CuSO_4 \rightleftarrows Cu^{++} + SO_4^{--}$$

Likewise, barium chloride solution contains the ions $Ba^{++}$ and $Cl^-$, $Cl^-$. Now, when a solution of barium chloride is mixed with any solution containing the $SO_4^{--}$ ion, the two ions $Ba^{++}$ and $SO_4^{--}$ unite to form the insoluble $BaSO_4$, which precipitates; hence the reaction proceeds to completion (page 336 of text). *The barium chloride test is therefore really a test for the presence of the sulfate ion* $SO_4^{--}$. Since only sulfuric acid and its salts give this ion, however, it is customary to say that it is a test for sulfuric acid and the sulfates.

*c. Normal sulfates: Crystallized copper sulfate.* In an evaporating dish place 7 g. copper oxide and 25 cc. water, and heat nearly to boiling. Add sulfuric acid (***Caution***) 2 or 3 drops at a time until the copper oxide has nearly all dissolved. Boil for a few minutes. Filter the solution into a clean beaker, cover the top of the beaker with a filter paper and set it aside to crystallize until the next laboratory period. Filter off the crystals of copper sulfate, dry them on filter paper and save them for Exercise 59, *a.*

*d. Acid sulfates: Crystallized potassium hydrogen sulfate.* The following directions illustrate the method by which the acid salt of dibasic acid may be prepared (page 261 of text).

Measure 15 cc. of concentrated sulfuric acid (sp. gr. 1.84), and (***Caution***) pour it a little at a time and with constant stirring into about 30 cc. of water. Divide the well-stirred solution into two equal parts. Calculate the weight of potassium hydroxide that will be required to neutralize one of these portions, dissolve it in as little water as possible, and add most of it slowly and with constant stirring to the one portion. Add the remainder more carefully, testing with litmus paper, until the solution is just neutral. (5) What is now present? To the neutral solution add the second portion of the sulfuric acid and evaporate the united solutions to about 40 cc. Set the beaker aside until the next laboratory period. Filter off the crystals and dry them on filter paper. (If only a few are present, evaporate the solution further for a second crop.) (6) Dissolve a crystal in water and test its reaction with litmus paper. Heat a few crystals in a crucible. (7) What is evolved?

## EXERCISE 59

## HYDRATES

**Object.** To study the properties of hydrates.

**Apparatus.** Burner; test tubes; porcelain crucible; ring stand; pipestem triangle; balance; evaporating dish.

**Materials.** 2 g. of zinc sulfate crystals; 6 g. of copper sulfate crystals; clear crystal of sodium sulfate.

*a. Hydrates.* Heat some small dry crystals of zinc sulfate in a dry test tube. (1) What evidence have you of the presence of water in the crystals? (2) Contrast the residue with the original crystals.

Select some small crystals of copper sulfate. (3) Do they appear to be dry? Fill a test tube one fourth full of these crystals, and heat until no further apparent change takes place. (4) Compare the residue with the original crystals in form, color, and composition. Dissolve the residue in as little hot water as possible, pour the solution (note its color) into an evaporating dish, and set aside until crystals are deposited. (5) Do these appear to be identical with the original crystals of copper sulfate in shape and color?

The water evolved when hydrates are heated is sometimes called water of crystallization. (6) Is the term appropriate? (7) Distinguish between the terms *hydrate, hydroxide, anhydrous, anhydride.*

*b. Efflorescence.* Expose a clear crystal of sodium sulfate to the air for one or two hours. (8) Explain the change in its appearance.

*c. Quantitative determination of the amount of water expelled on heating the hydrate of copper sulfate.* Weigh accurately (to 0.01 g.) a porcelain crucible and cover, recording the weight in the table below. Then add 2 or 3 g. of crystals (no larger than a pea) of the hydrate of copper sulfate and again weigh accurately. Place the covered crucible on a pipestem triangle (Fig. 30) and heat it with a gentle flame until the crystals lose their color. This will require from twenty to thirty minutes. The tip of the flame should not quite touch the crucible. The product is anhydrous copper sulfate. When the crucible is cool, reweigh and record the weight. Make the calculations indicated below:

### Table of Results

1. Weight of crucible and cover . . . . . . . . . . . . . . g.
2. Weight of crucible, cover, and hydrate . . . . . . . . . g.
3. Weight of hydrate taken (Calculate) . . . . . . . . . . g.
4. Weight of crucible, cover, and anhydrous copper sulfate . g.
5. Weight of anhydrous copper sulfate (Calculate) . . . . . g.
6. Weight of water in the hydrate (Calculate) . . . . . . . g.
7. Percentage of water in the hydrate (Calculate) . . . . . %

(9) The formula of the hydrate being $CuSO_4 \cdot x\,H_2O$, calculate the number of molecules of water present in each molecule of the hydrate.

## EXERCISE 60

## SOME COMPOUNDS OF PHOSPHORUS

**Object.** To prepare some of the compounds of phosphorus and to study their properties.

**Apparatus.** 250-cc. wide-mouthed bottle; deflagrating spoon; glass plate; small beaker; porcelain crucible; burner; evaporating dish; stirring rod; test tube.

**Materials.** 0.5 cc. of phosphorus trichloride; phosphorus (size of a pea); litmus paper (red and blue); 10 cc. of ammonium molybdate solution (R.S.); ammonium hydroxide; nitric acid; silver nitrate solution (R.S.); hydrochloric acid; 5 g. of disodium phosphate; 2 g. of sodium ammonium phosphate.

*a. Phosphorous acid.* Into a test tube pour about 0.5 cc. of phosphorus trichloride (***Hood***) and add a little water, a drop at a time. Mix the liquids by shaking the tube. Test the gas evolved by holding a strip of blue litmus at the mouth of the tube; also by holding in the mouth of the tube the end of a glass rod previously dipped into concentrated aqua ammonia. (1) Write the equation for the reaction between phosphorus

trichloride and water. Finally add about 5 cc. of water to the tube, pour the liquid into an evaporating dish, and evaporate to a thick sirup. Dilute this with a little water; transfer the solution to a test tube and add a few drops of a solution of silver nitrate. Boil the resulting mixture; finely divided metallic silver (black) precipitates. (2) Explain.

*b. Phosphoric acid.* Cover the bottom of a wide-mouthed bottle (250-cc.) with water to a depth of about 1 cm. Place a piece of phosphorus on a deflagrating spoon and ignite it by touching it with a hot wire. Quickly lower the phosphorus into the bottle and cover the mouth of the bottle with a glass plate. When the phosphorus ceases to burn, withdraw the spoon and allow the fumes in the bottle to dissolve in the water. (3) Test the solution with litmus paper and account for the results.

Pour the solution into a small beaker, add 2 or 3 cc. of nitric acid, and boil the solution until about half of it evaporates. All the phosphorus compounds present are oxidized to phosphoric acid by the nitric acid. Add a few drops of the solution to 10 cc. of a solution of ammonium molybdate and warm gently. Add ammonium hydroxide to the mixture until the liquid is alkaline. Again acidify the liquid with nitric acid. (4) Note all the results. *The formation of a yellow precipitate upon the addition of ammonium molybdate, which precipitate is soluble in ammonium hydroxide but insoluble in nitric acid, serves as a good test for phosphate ions* ($PO_4^{---}$).

Ammonium molybdate is a complex compound, having the formula $(NH_4)_6Mo_7O_{24} \cdot 4\,H_2O$ (see page 702 of text). The yellow precipitate formed when ammonium molybdate reacts with phosphoric acid or a phosphate is a very complex compound (for formula, see page 702 of text) commonly known as ammonium phosphomolybdate.

*c. Pyrophosphates.* Apply a gentle heat to 1 or 2 g. of disodium phosphate placed in a porcelain crucible. Gradually increase the heat to the full extent of the Bunsen burner, and continue the heating for about 10 minutes. (5) What is the effect of heat on monohydrogen phosphates (page 413 of text)? When the crucible has cooled dissolve the product in cold water, and test the solution with silver nitrate solution. Normal silver pyrophosphate is precipitated. (6) Give its formula and color. (7) Compare with the precipitate formed ($Ag_3PO_4$) by adding silver nitrate solution to a solution of disodium phosphate which has not been heated.

*d. Metaphosphates.* Heat 1 or 2 g. of sodium ammonium phosphate (microcosmic salt, page 413 of text) as in *c*. This salt, when heated, acts as does the monosodium salt. Test the product with a solution of silver nitrate as in *c*. Silver metaphosphate is precipitated. (8) Give its formula and color. (9) Contrast the action of silver nitrate on orthophosphates, pyrophosphates, and metaphosphates.

## EXERCISE 61

### ARSENIC AND ITS COMPOUNDS

**Object.** To study the properties of arsenic and some of its compounds.

**Apparatus.** Blowpipe; piece of charcoal (8 × 3 × 2 cm., Fig. 76); hard-glass tube (10 cm. long and 6 or 7 mm. wide); file; burner; porcelain crucible; test tube; hydrogen generator and tubes as shown in Fig. 74 (the tube *B* is filled with calcium chloride and *C* is a hard-glass tube drawn out to a jet at *D*); hydrogen sulfide generator.

**Materials.** Arsenic (size of a grain of wheat); 0.2 g. of arsenious oxide; 2 or 3 g. of charcoal (pieces the size of a grain of wheat); zinc and dilute sulfuric acid for generating hydrogen; iron sulfide and hydrochloric acid for generating hydrogen sulfide.

***a.*** Practice the use of a blowpipe as explained in ***e***, Exercise 70, until you can produce a continuous and steady flame. Then place a bit of arsenic in a cavity on a piece of charcoal and gently heat it in the tip of the blowpipe flame (***Hood***). Note the peculiar, garlic-like odor (*poisonous*).

***b.*** Introduce into a hard-glass tube an amount of arsenious oxide equal in bulk to a grain of wheat. Cover this to a depth of 2 or 3 cm. with somewhat finely powdered charcoal which has been previously heated to a high temperature in a porcelain crucible. See that the inner surface of the tube above the charcoal is perfectly clean.

Incline the tube and heat the *upper* portion of the charcoal to a high temperature; then, while maintaining the charcoal at this temperature, gradually bring the lower part of the tube also into the flame. The upper part of the tube must be kept as cool as possible. The arsenious oxide is changed into a vapor, which passes over the hot charcoal. Note the coating formed on the wall of the upper part of the tube. (1) Account for the result.

Cut the tube as near the bottom as possible and remove the charcoal; then, inclining the tube, apply a very *gentle* heat to that portion of it which contains the coating. Note that small white crystals are slowly deposited in the colder portions of the tube. (2) What is the compound? Examine the crystals with a magnifying glass. (3) Note their form.

***c.*** In a test tube, place a bit of arsenious oxide equal in bulk to a grain of wheat. Add 2 or 3 cc. of hydrochloric acid and an equal volume of water to the tube and heat slightly until the oxide dissolves. Now pass a few bubbles of hydrogen sulfide through the solution. (4) Account for the results.

*Caution. Perform the following experiment in the* ***hood,*** *as the arsine formed is very poisonous.*

*d. Marsh's test.* Arrange an apparatus according to Fig. 74. Generate hydrogen in *A* by the usual method, and, *after taking the usual precautions*, ignite it as it escapes from the glass jet. Add sufficient acid from time to time to cause a gentle evolution of the gas. Now apply strong heat to the hard-glass tube *C* at a place near its center, using the "wing-top" burner. After a few minutes note whether any deposit forms just beyond the heated portion of the tube. If none forms, the materials are free from arsenic. Now add 2 or 3 drops of a dilute hydrochloric acid solution of arsenious oxide (made by dissolving a bit of the oxide no larger than a pinhead in 1 cc. of the dilute acid) to the generator, rinsing it down the funnel tube with a little water. Continue the heating of the hard-glass tube at the same place. Note the deposit formed on the sides of the tube. Withdraw the heat and thrust a crucible cover into the flame of the burning hydrogen for 2 or 3 seconds. A black deposit of arsenic forms. Cut the tube *C* containing the deposit so as to remove only the jet and, inclining it, apply a *gentle* heat, as in *b*. (5) Account for the results and write the equations for all the reactions involved in the experiment.

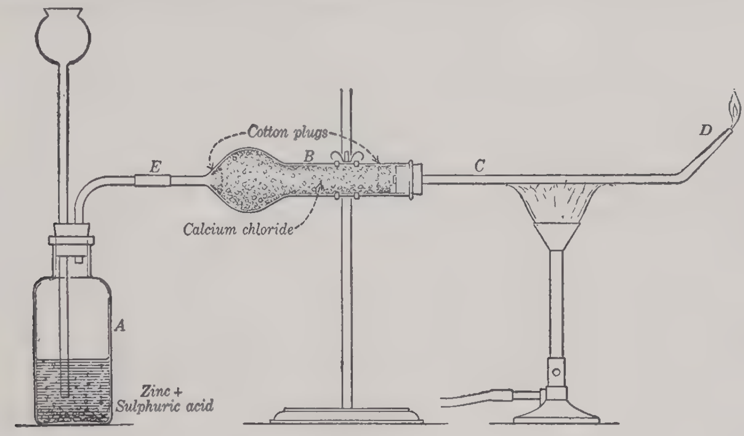


FIG. 74

## EXERCISE 62

## ANTIMONY AND ITS COMPOUNDS

**Object.** To study the properties of antimony and of some of its compounds.

**Apparatus.** Blowpipe; beaker; stirring rod; hydrogen sulfide generator; test tube.

**Materials.** Piece of charcoal (8 × 3 × 2 cm.); 2 pieces of antimony (the size of a grain of wheat); hydrochloric acid; nitric acid; strip of zinc; 10 g. of ferrous sulfide.

*a.* Heat a bit of antimony on charcoal as in *a*, Exercise 61. The product is $Sb_2O_3$. (1) Write the equation for its formation.

*b.* Introduce into a test tube a bit of antimony no larger than a grain of wheat and add about 3 cc. of hydrochloric acid and then 2 or 3 drops of nitric acid. $SbCl_3$ is formed. After the metal has dissolved, pour the solution into 50 cc. of water. (2) Account for the formation of the white solid (page 421 of text; see also *b*, Exercise 46). Now add hydrochloric acid, drop by drop, with constant stirring, until the precipitate dissolves. Half fill a test tube with the resulting solution and insert a strip of zinc. (3) Note the results (compare Exercise 40). Through the remainder of the solution pass a few bubbles of hydrogen sulfide. The product is $Sb_2S_3$. (4) Note its properties and write the equation for its formation.

## EXERCISE 63

## BISMUTH AND ITS COMPOUNDS

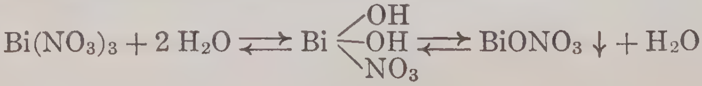
**Object.** To study the properties of bismuth and of some of its compounds.

**Apparatus.** Blowpipe; beaker; stirring rod; hydrogen sulfide generator (Fig. 72).

**Materials.** 2 pieces of bismuth (size of a grain of wheat); piece of charcoal (8 × 3 × 2 cm.); nitric acid; 10 g. of ferrous sulfide; hydrochloric acid.

*a.* Heat a piece of bismuth on charcoal, as in the case of antimony (Exercise 62). $Bi_2O_3$ is formed and is deposited as a coating on the charcoal. (1) Summarize the effects of heating arsenic, antimony, and bismuth in air.

*b.* Introduce a bit of bismuth into a test tube, add about 5 cc. of nitric acid, and warm gently until the bismuth is dissolved. Bismuth nitrate ($Bi(NO_3)_3$) is formed. Pour the resulting solution into a beaker containing 50 cc. of water. A white precipitate of bismuth subnitrate is formed in accordance with the following equation.



$$Bi(NO_3)_3 + 2\,H_2O \rightleftarrows Bi\begin{matrix}OH\\OH\\NO_3\end{matrix} \rightleftarrows BiONO_3\downarrow + H_2O$$

Stir the mixture and divide it into two parts. To part one add nitric acid, a drop at a time with stirring, until the liquid clears. (2) Explain. Through the other part pass a few bubbles of hydrogen sulfide. (3) Explain. (4) Contrast the action of hydrogen sulfide on salts of antimony (see Exercise 62) and of bismuth.

## EXERCISE 64

## COMPOUNDS OF SILICON

**Object.** To study the properties of some of the compounds of silicon.

**Apparatus.** Small iron crucible; evaporating dish; small beaker; ring stand; burner; funnel and filter paper.

**Materials.** 0.5 g. very fine sand; 2 g. solid sodium hydroxide; 2 cc. of water glass; hydrochloric acid.

*a.* In a small iron crucible place about half a gram of very finely divided sand together with about 2 g. of solid sodium hydroxide. Heat the crucible in a hot flame until the mixture melts and again becomes solid. Cool the crucible and contents and then dissolve the contents of the crucible in about 20 cc. of hot water. (1) Write the equation for the reaction on the assumption that sodium orthosilicate is formed. Add a few drops of hydrochloric acid to the solution. (2) Explain the results, writing the equation for the reaction.

*b.* (3) What is water glass (page 433 of text)? Pour 2 or 3 cc. of water glass into an evaporating dish and dilute it with 10 cc. of water. Add to the liquid 3 or 4 cc. of hydrochloric acid. Note the formation of a gelatinous precipitate. (4) Account for its formation, assuming that water glass is a solution of sodium metasilicate. Now evaporate the mixture to dryness and then heat the solid residue gently with a bare flame. (5) What is the residue? (6) Account for its formation. When the dish is cool, add 10 cc. of water, stir, and filter. (7) Note the properties of the solid on the filter paper.

*c.* (8) Recall the action of hydrofluoric acid on silica (Exercise 47).

# EXERCISE 65

## COMPOUNDS OF BORON

**Object.** To study the properties of some of the compounds of boron.

**Apparatus.** Fine platinum wire (a piece 5 cm. long fused in a glass tube *A* for a handle, as shown in Fig. 75); beaker (100-cc.); stirring rod; funnel and filter paper; medicine dropper; small crucible; burner.

**Materials.** 8 g. of borax; sulfuric acid; 1 or 2 drops of cobalt nitrate solution (R.S.).

*a. Borax.* This is the most important of the compounds of boron. (1) Note carefully its physical properties. Make a little loop on the end of a platinum wire and heat it to redness in the tip of a Bunsen flame (Fig. 75); then quickly bring the hot loop in contact with some borax and reheat in the tip of the flame (Fig. 75). The borax adhering to the loop will swell up (owing to the expulsion of the water of hydration) and finally form a clear, glassy bead. If the bead is very small, bring the hot bead into contact with more borax and again heat until the bead is clear.

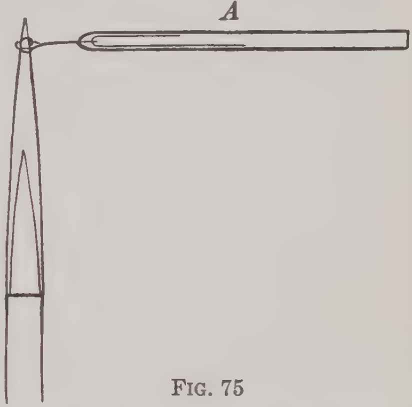


FIG. 75

Moisten the bead with a drop of a solution of any cobalt compound and reheat until the bead is transparent when cold. (2) Note the color of the bead now (the depth of color depends upon the amount of cobalt present). This property serves as a simple test for cobalt. Some of the other metals likewise impart characteristic colors to the bead, as will be explained later. This method for detecting metals is known as the *borax-bead test.*

*b. Boric acid.* Dissolve 5 g. of borax in 15 cc. of boiling water. *Carefully* add (drop by drop) to the hot solution 2 or 3 cc. of sulfuric acid and stir. The sulfuric acid acts on the borax as follows:

$$Na_2B_4O_7 + H_2SO_4 + 5\ H_2O \longrightarrow Na_2SO_4 + 4\ H_3BO_3 \downarrow$$

The boric acid ($H_3BO_3$) is insoluble and separates as a precipitate. Filter off the precipitate; then place a bit of it on the loop of the platinum wire used in *a*, above, and (3) note the color that it imparts to the flame. Transfer the boric acid to a small crucible and gradually heat to a high temperature. (4) What compounds are formed as the temperature rises, and what is the final product (page 437 of text)?

*c. Test for borax.* Make a borax bead as in *a.* (5) What color does the borax impart to the flame? Now dip the end of a glass rod into sulfuric acid, moisten the bead with the acid clinging to the end of the rod, and then touch the bead to the edge of the flame. (6) Note and account for the results. This serves as a simple test for borax.

## EXERCISE 66

## COLLOIDAL SYSTEMS

**Object.** To prepare certain colloidal systems and to study their properties.

**Apparatus.** Test tubes; funnel and filter paper; 3 beakers; burner.

**Materials.** 1 g. of sulfur; 5 cc. of alcohol; 3 drops of a solution of auric chloride prepared by dissolving 0.3 g. of the chloride in 50 cc. of water; 1 cc. of formalin diluted with 10 cc. of water; 0.2 g. of antimony oxide; hydrochloric acid; 10 cc. of saturated solution of hydrogen sulfide; 0.1 g. of tartar emetic; ferric chloride (R.S.); 2 g. of sodium acetate; 1.5 g. of ammonium chloride; sulfuric acid; 1 g. of agar; 50 cc. of litmus solution; 10 g. of bone-black; bit of white soap; 5 cc. of kerosene; 10 cc. of water glass, density 1.1.

*a. Preparation of colloidal systems.* Prepare 4 or 5 cc. of a saturated solution of sulfur in alcohol and pour it into 100 cc. of water. A colloidal system is thereby formed. (1) In this system what is the dispersed phase and what is the dispersing phase? Filter some of the mixture. (2) Is the sulfur removed by filtration?

To 10 cc. of water in a test tube add 3 drops of the dilute solution of auric chloride. Heat to boiling and add 2 cc. of the dilute solution of formalin (page 491 of text), which acts as a reducing agent. Set the solution aside for five minutes, noting the changes in color. The gold is liberated, forming the dispersed phase of a colloidal system. The color is due to the metallic gold. (3) Note the color, comparing the appearance when viewed by transmitted light with that viewed by reflected light.

*b. Preparation of colloidal systems by double decomposition.* Prepare a little antimony sulfide ($Sb_2S_3$) by dissolving a small bit of antimony oxide the size of 1 or 2 wheat grains in 5 cc. of dilute hydrochloric acid and adding slowly 10 cc. of a saturated solution of hydrogen sulfide. After the precipitate has settled, (4) note the color of the supernatant liquid. Set the tube containing it aside for reference.

Dissolve a little tartar emetic (this contains antimony as a salt of a *weak* acid), the size of 2 or 3 wheat grains, in about 100 cc. of water and add slowly, with stirring, 10 cc. of the saturated solution of hydrogen sulfide. (The resulting solution should not have the odor of hydrogen sulfide.) (5) What change do you notice? Filter a few cubic centimeters of the solution. (6) Is the filtrate colorless? (7) Is a precipitate left on the filter? Boil 2 or 3 cc. of the filtrate and again filter. (8) Does the heat cause the antimony sulfide to separate? Designate the liquid as A and reserve for tests below.

*c. Preparation of colloidal systems by hydrolysis.* To 90 cc. of water in a beaker add 10 cc. of the ferric chloride solution. Slowly heat the solution to boiling and (9) note the change in color. The salt is hydrolyzed with formation of colloidal hydrated ferric oxide. (10) View the color by reflected light and then by transmitted light. Designate the liquid as B and save for further tests.

Dissolve 2 grams of sodium acetate (this is the sodium salt of the weak acetic acid) in 50 cc. of water, and add to it 1 cc. of a solution of ferric chloride. (11) What new salts should you expect would form? (12) Should either of these give extensive hydrolysis? (13) Is a precipitate obtained? (14) What is the color of the liquid? The color is due largely to the presence of colloidal hydrated ferric oxide. Designate the liquid as C and save for further tests.

*d. Coagulation of colloidal systems.* Heat to boiling 5 cc. of the colloidal systems designated above as A, B, and C. (15) Does the dispersed phase precipitate? Add about half a gram of solid ammonium chloride to 5 cc. of A, B, and C, respectively, and shake the mixtures vigorously. (16) Does a precipitate form in any of the liquids? To 5 cc. of each add 1 drop of concentrated sulfuric acid. Shake the solutions and (17) note and explain the results.

*e. Protecting colloids.* Dissolve 0.1 g. of agar (a colloidal material resembling gelatin) in about 10 cc. of hot water. Add equal portions of this solution to portions of the colloidal systems designated above as A, B, and C. With these solutions repeat the experiments of *d*. (18) Does the presence of the agar interfere with coagulation? Organic colloids often interfere with precipitation tests in chemical analysis. (19) What are such colloids called?

*f. Adsorption.* Fold a small filter to fit a funnel and place upon it a layer of boneblack, 2 or 3 cm. thick. Wet it thoroughly with water and let it drain. Prepare 50 cc. of a solution of blue litmus, Congo red, or some other dye, using just enough of the dye to make the solution decidedly colored. Slowly filter the colored solution through the boneblack, repeating several times. (20) Is the color removed?

*g. Emulsions.* Select two test tubes of the same size. Place in the bottom of one to a depth of about 2 cm. some *thin* pieces of white soap cut from a bar of soap; then fill the tube about two thirds full of distilled water. Warm the mixture and shake it gently until the soap dissolves. Now fill the other tube about two thirds full of water. Next pour into each tube about 2 cc. of kerosene. Note that the oil floats on the top of the water in each tube.

Now take a tube in each hand, close the mouth of the tube firmly with your thumb, and shake the tube and contents vigorously for

one minute. (21) Note the appearance of the liquids in each tube. (22) What is such a mixture called? Set the tubes aside for five or ten minutes (or until the next laboratory period) and again examine the liquids in the tubes. (23) Have the oil and water separated in both tubes? (24) What term is applied to a substance, such as soap, that prevents or at least delays the separation of the liquids in an emulsion?

*h. Gels.* Break up some dry gelatin or agar into very small pieces and place them in a test tube to a depth of about 2 cm. Add 10 cc. of water and heat nearly to boiling, until a clear solution is obtained. Immerse the test tube in ice water for a time. (25) What change do you note? Again put the tube into hot water, and again cool, and (26) note the results.

To about 10 cc. of water glass of density 1.1 add 10 drops of sulfuric acid. Shake the solution thoroughly and set aside if necessary until the next period. (27) Note and explain the results. Tap the test tube gently and note the vibrations of the gel.

## EXERCISE 67

### CARBONIC ACID; THE CARBONATES; UREA; THE THIOCARBONATES

**Object.** To study the reactions of some important carbon compounds.

**Apparatus.** Test tubes; test-tube rack; glass rod; 60-cc. bottle and 2-hole stopper; hydrogen generator; delivery tube; large test tube; clamp; pipette; funnel; filter paper; ring stand and burner; small beakers.

**Materials.** 3 g. of sodium hydroxide; 15 g. of marble; 0.5 g. of sodium carbonate; 3 or 4 cc. solutions of calcium chloride, copper sulfate, zinc sulfate, ferrous sulfate; lime water; 0.7 g. of urea; 10 cc. solution of sodium hypobromite (see Appendix for preparation); 16 g. of crystallized barium hydroxide; 15 g. of ferrous sulfide; 1 cc. of carbon disulfide; 15 cc. of denatured alcohol; hydrochloric acid; sodium hydroxide.

*a. Carbonic acid.* In a test tube place about 0.5 g. of sodium carbonate and set the tube in your rack. Now add a few drops of hydrochloric acid to the dry salt. (1) What would you expect to be formed? (2) How do you account for the effervescence? Treat a small piece of marble and any other available carbonates in the same way, preserving the test tubes in your rack. Dip a clean glass rod into clear lime water and carefully insert the moistened end into the mouth of the test tube that contained sodium carbonate (Fig. 46). (3) How do you account for the result? Test the other tubes in the same way. This procedure constitutes a convenient test for carbonates.

*b. The carbonates.* Dissolve about 3 g. of sodium hydroxide in 30 cc. of water and pour the solution into your 60-cc. bottle. In a hydrogen generator (Fig. 34) put about 15 g. of marble in small pieces, with enough water to completely cover the solid, and provide the generator with a delivery tube to reach to the bottom of the solution of sodium hydroxide. Through the funnel tube add dilute hydrochloric acid in small portions at a time so as to maintain a steady but not too rapid current of carbon dioxide. From time to time withdraw the delivery tube from the small bottle, stopper the bottle with the palm of your hand, and shake it vigorously to assist in the absorption of the gas. Continue the process until all the marble has dissolved. (4) What should be the first product of the reaction? (5) How should this act on excess of carbonic acid? Pour the solution into an evaporating dish and heat it slowly to boiling. (6) What gas is evolved? (7) How do you account for this? Boil until the gas ceases to be evolved. To 5 cc. of the solution in a test tube add a few drops of hydrochloric acid. (8) Write the equation for the reaction. Obtain 3 or 4 cc. of solutions of some salt of calcium, of copper, of zinc, and of nickel (all of which metals are bivalent). Test each solution with a few drops of the solution in the evaporating dish. (9) On the assumption that the normal carbonate is precipitated, write the equations for the reactions. (10) What carbonates are soluble (page 453 of text)?

*c. Urea.* Dissolve about 0.5 g. of urea in 2 cc. of water, add 5 cc. of sodium hydroxide, and heat to boiling in a test tube. (11) Can you detect the odor of ammonia? (12) Account for its formation. Place 10 cc. of the solution of sodium hypobromite (R. S.) in a large test tube held vertically by a clamp on your ring stand. Draw into a pipette 3 or 4 cc. of a solution of urea prepared by dissolving 0.2 g. in 5 cc. of water. Keeping your finger on the top of the pipette, dip the delivery point of the pipette to the bottom of the solution in the test tube, then carefully force *nearly all* the urea into the hypobromite by gently blowing into the top of the pipette, being careful not to completely empty the pipette nor to blow any bubbles of air into the solution of hypobromite. (13) What is the most characteristic chemical reaction of hypochlorites and hypobromites (pages 370–374 of text)? (14) The equation

$$2\,CO(NH_2)_2 + 3\,O_2 \longrightarrow 2\,N_2\uparrow + 2\,CO_2\uparrow + 4\,H_2O$$

expresses the reaction of a hypobromite together with an excess of alkali on urea. What gas collects in the inverted test tube?

*d. Preparation of barium thiocarbonate.* Dissolve 16 g. of crystallized barium hydroxide ($Ba(OH)_2 \cdot 8\,H_2O$), in 50 cc. of warm water, and divide the solution into two equal parts. Provide the 60-cc. bottle with a 2-hole stopper, and through one hole insert a delivery tube reaching to the bottom of the bottle. Partly close the other hole by a short piece of glass tubing drawn out to a jet. In the bottle pour half the solution of barium hydroxide (which is very much like calcium hydroxide in chemical action but is more soluble). Charge your hydrogen

generator with about 15 g. of iron sulfide in small pieces, cover these with water, and connect the delivery tube of the generator with the tube extending to the bottom of the small bottle containing the barium hydroxide. Place the whole apparatus in the hood. Add hydrochloric acid to the gas generator in small portions at a time so as to maintain a slow but steady current of hydrogen sulfide, and continue the flow of this gas until no more is absorbed by the barium hydroxide. (The glass jet in the stopper of the small bottle is to keep the gas from escaping too rapidly, and so to promote absorption.) (15) What is now present in the solution (recall *a* of this exercise)? Disconnect the small bottle and pour its contents into the other half of the solution of barium hydroxide. (16) What is now present in solution? To this solution add 1 cc. of carbon disulfide, and shake the solution vigorously. The yellow precipitate is barium thiocarbonate ($BaCS_3$), the sulfur analogue of barium carbonate ($BaCO_3$). (17) Write the equation for its formation. Filter off the precipitate, wash it on the filter first with water and then with a little denatured alcohol. Dry it on filter paper in a warm place. (18) How should it act when treated with an acid?

## EXERCISE 68

### HYDROCARBONS

**Object.** To prepare some of the most common hydrocarbons and to study their properties.

**Apparatus.** Evaporating dish; burner; hard-glass tube fitted with a cork and a delivery tube, as used in the preparation of oxygen; two 250-cc. bottles; pneumatic trough; watch glass; beaker; stirring rod; test tubes; mortar and pestle.

**Materials.** 15 g. of soda lime (mixture of sodium hydroxide and calcium hydroxide); 10 g. of fused sodium acetate; wooden splints; iron wire; bit of calcium carbide (size of a bean); sulfuric acid; sodium hydroxide; 10 cc. each of kerosene and low-boiling gasoline; 1 g. of paraffin.

*a. Methane.* Mix intimately, by grinding together in a mortar, 15 g. of soda lime and 10 g. of fused sodium acetate. Transfer the mixture to the hard-glass tube used in preparing oxygen and proceed just as in the preparation of oxygen, except that the tube should be clamped in a horizontal position while being heated. Before heating the tube it is well to wrap a piece of wire gauze around it as a protection in case it should break. Collect over water one or two bottles of the evolved gas. (1) Note the color and odor of the gas. (2) Is it inflammable?

Sodium acetate has the formula $NaC_2H_3O_2$. When heated with the sodium hydroxide or soda lime, methane is generated according to the following reaction:

$$NaC_2H_3O_2 + NaOH \longrightarrow Na_2CO_3 + CH_4\uparrow$$

*b. Acetylene.* Nearly fill a test tube with water and drop into it a small piece of calcium carbide. Note the gas evolved. (3) Write the equation for its formation. (4) Ascertain whether the gas is inflammable by holding a lighted splint at the mouth of the tube.

*c.* Obtain from the instructor a few drops each of gasoline and of kerosene. Test the inflammability of each by dipping the end of a glass rod in the liquid and then touching it to the tip of a flame. Place 10 drops of gasoline (***Caution.*** *Keep away from all flames*) on a watch glass (***Hood***) and set the glass on a beaker half filled with boiling water. (5) Note the time required for its complete evaporation. (6) Repeat the experiment, using 10 drops of kerosene. (7) Which of the two substances is the more volatile?

*d.* Pour 2 or 3 drops of gasoline into a warm 250-cc. wide-mouthed bottle. Cork the bottle and shake it vigorously. Now remove the cork and, standing at arm's length, bring a lighted splint to the mouth of the bottle. (8) Account for the results. (9) What use is suggested by the property noted in this experiment?

*e.* Study the properties of paraffin. (10) Is it soluble in water? in kerosene? (11) Will it melt at the temperature of boiling water? (12) Will it burn? (13) Test the action of acids and alkalies on paraffin by adding a few drops of each to a bit of the paraffin on a watch glass. (14) What properties are suggested by the term *paraffin* (see derivation of word)?

## EXERCISE 69

## SOME DERIVATIVES OF METHANE

**Object.** To study the properties of chloroform, carbon tetrachloride, and iodoform.

**Apparatus.** Test tubes; beaker (100-cc.); glass rod.

**Materials.** 2 cc. of chloroform; 1 cc. of cottonseed oil; 3 cc. of carbon tetrachloride; splint; 2 cc. of alcohol; 5 cc. of iodine solution (R.S.); sodium hydroxide.

*a. Chloroform.* (1) Note its odor. Add about 1 cc. of chloroform to an equal volume of water. (2) Does it mix with water? (3) Is it heavier or lighter than water? Add 2 drops of an oil (such as cottonseed oil) to 2 cc. of chloroform. (4) Is the oil dissolved by the chloroform? (5) Determine whether chloroform will burn by bringing a drop on the end of a glass rod into the edge of a flame for an instant.

*b. Carbon tetrachloride.* (6) Repeat all the experiments listed under chloroform, substituting carbon tetrachloride for the chloroform. (7) Pour about 2 cc. of carbon tetrachloride into a small beaker (100-cc.) and heat it until the vapor of the liquid fills the beaker; then thrust a lighted splint into the vapor. Mention two important commercial uses of carbon tetrachloride illustrated in the above experiments.

*c. Iodoform.* Pour about 2 cc. of alcohol into a small beaker and add 5 cc. of iodine solution. Now add to the mixture a solution of sodium hydroxide, drop by drop, stirring the liquid after each addition. Continue until the color of the iodine is completely discharged. Note the formation of a yellowish solid (iodoform). (8) Note its odor. (9) What is the chief use of the compound?

## EXERCISE 70

### FLAMES

**Object.** To study the properties of flames.

**Apparatus.** Wire gauze; porcelain dish; blowpipe; burner.

**Materials.** Charcoal (size of a bean); 5 cc. of limewater (R.S.); candle; wooden splint; piece of charcoal (8 × 3 × 2 cm.); bit of lead as large as a pea; 2 g. of lead oxide.

*a. Parts of a flame.* (1) Note and account for the difference between the combustion of a wooden splint and that of a piece of charcoal. (2) What are the conditions necessary for the production of a flame? Light a candle and place it so that the flame is against a black background and is not disturbed by air drafts; then (3) note the different cones in the flame. Test the relative temperatures of different parts of the flame by means of narrow strips of splints. (4) Draw a diagram in your notebook, showing the different parts of the flame and noting the color of each of the cones. Extinguish the candle flame and hold a lighted splint 2 or 3 cm. from the wick in the little column of smoke rising from the wick. (5) Note and account for the result.

*b. Products of combustion.* (6) What two elements constitute the main percentage of ordinary fuels? (7) What products form when these elements burn in air or in oxygen? (8) Devise and describe simple experiments to show the presence of these products in the gases evolved by the burning candle.

*c. Kindling temperature.* (9) What is meant by the kindling temperature of gases? When an oil lamp is first lighted, a film of liquid often spreads over the chimney for an instant. (10) Explain. Press a piece of wire gauze halfway down on a Bunsen flame. Notice that the flame does not extend above the gauze. (11) Is this due to the absence there of gases that are combustible (test for their presence by means of a lighted splint)?

Turn off the gas, then turn it on and ignite it over a piece of wire gauze held horizontally 4 or 5 cm. above the top of the burner. (12) Note the results and explain. (13) How does the miner's safety lamp prevent explosions?

*d. Luminous flames.* Hold a porcelain dish in a candle flame, also in a small luminous Bunsen flame. (14) Account for the deposition of carbon. (15) Does the nonluminous flame deposit carbon? (16) To what is the luminosity of the flame due?

*e. Use of the blowpipe.* Close the openings on the Bunsen burner so as to secure a luminous flame; then lower the flame until about 3 cm. high. Now place the end of the blowpipe just inside the lower part of the flame and blow gently through the blowpipe (Figs. 76, 77). The flame will be bent over, as shown in the figures. Practice this until you can produce a *continuous* and *steady* flame by forcing a portion of the exhaled air through the blowpipe by constant tension of the muscles of the cheek. It will be noted that the flame so produced consists of two cones, one inside the other. The inner cone, being rich in carbon monoxide, is a *reducing* flame (Fig. 77); the outer cone, being very hot and in contact with the oxygen of the air, is an *oxidizing* flame.

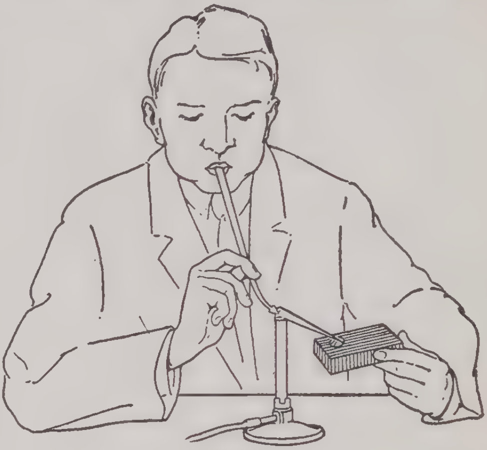


FIG. 76

Make a shallow cavity near the end of a piece of charcoal and place in this a bit of lead. Now heat this in the oxidizing flame as shown in Fig. 76. The lead is changed to lead oxide (PbO), a yellow coating which collects about the cavity on the surface of the charcoal.

Clean the charcoal and place in the cavity a paste of lead oxide (PbO) made by adding a little water to 2 g. of the oxide and mixing. Now heat this in the reducing flame (hold the charcoal so that the oxide is in the tip of the inner cone). The oxide will be gradually reduced to a bead of metallic lead.

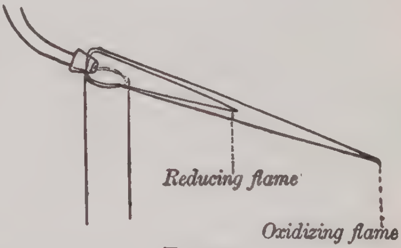


FIG. 77

NOTE. Save the charcoal for future experiments, but moisten it with water to extinguish any fire present. Indeed, to avoid all danger from fire it would be better if the charcoal used by the students were kept in some noncombustible vessel, such as a crock or pneumatic trough.

## EXERCISE 71

## THE CARBOHYDRATES

**Object.** To study some of the properties of the sugars and starches.

**Apparatus.** 3 test tubes; 2 small beakers; stirring rod; burner; microscope.

**Materials.** 3.5 g. of copper sulfate crystals dissolved in 50 cc. of water (label this solution A); 17.5 g. of sodium potassium tartrate (Rochelle salts) dissolved in 42 cc. of the sodium hydroxide solution on your desk (label this solution B; solutions A and B should be poured into bottles and reserved for future exercises); 5 cc. of commercial glucose; 2 g. of sucrose; 1 g. each of sweets, such as candy, honey, and molasses; 2 or 3 g. of sodium carbonate dissolved in as little water as possible; red litmus paper; hydrochloric acid; iodine solution (R.S.); 2 g. of starch; 3 g. of sodium carbonate dissolved in a little water; starch solution (R.S.)

### The Sugars

*a. The test for dextrose.* The most common test for dextrose is the reaction with Fehling's solution. This is prepared as needed by mixing equal volumes of solutions A and B, prepared as directed above.

Pour into a test tube about 3 cc. each of solutions A and B. When thoroughly mixed, the resulting solution should be deep blue but perfectly clear. Heat the blue solution nearly to boiling, add 1 or 2 drops of commercial glucose, and continue the heating for a few moments. The copper sulfate in the solution is reduced to cuprous oxide by the dextrose, and this separates in the form of a red or yellow solid. Levulose will act in the same way. Dissolve samples of candy, honey, and molasses in a little water and test for the presence of dextrose and levulose in these sweets.

*b. The action of sucrose on Fehling's solution.* In a similar way try the action of pure sucrose on Fehling's solution. (1) Note the result.

Now dissolve about 1 g. of sucrose in 10 cc. of water. Add 4 or 5 drops of concentrated hydrochloric acid and slowly heat nearly to boiling. Set the solution aside for about five minutes, then cool and neutralize the acid present by adding a saturated solution of sodium carbonate until the resulting mixture is just alkaline to litmus paper. Now test this with Fehling's solution as in *a*. (2) Account for the result.

### Starch

*c. Microscopic appearance.* Examine under the microscope the appearance of starch from different sources (corn, wheat) when magnified. Draw diagrams of the starch granules.

*d. Actions of acids on starch.* Try the action of starch solution on Fehling's solution (as in *a*, above). (3) Record the result.

Add 2 cc. of hydrochloric acid to 50 cc. of starch solution in a beaker and boil the contents gently for thirty minutes, allowing the solution to concentrate to about 25 cc. Cool the liquid, neutralize with sodium carbonate, and again test the solution with Fehling's solution. (4) Note and account for your results.

*e. Test for starch.* Recall the action of iodine on starch (Exercise 50). This constitutes a good test for starch.

## EXERCISE 72

### ALCOHOLS; ALDEHYDES

**Object.** To study the properties of the two common alcohols; also of a common aldehyde.

**Apparatus.** Test tubes; small evaporating dish; beaker; stirring rod.

**Materials.** 7 or 8 cc. of ethyl alcohol; 5 cc. of methyl alcohol; small amounts (0.5 g.) of sucrose, sodium chloride, sulfur, and cottonseed oil; 5 cc. of limewater; iodine solution (R.S.); sodium hydroxide; 3 cc. of formalin; 6 cc. of Fehling's solution (see "Materials," Exercise 71); 10 cc. of a solution prepared by dissolving 20 g. of silver nitrate in 100 cc. of water and then adding ammonium hydroxide, a drop at a time, with constant stirring, until the precipitate first formed is *nearly* all dissolved.

*a. The properties of ethyl alcohol.* (1) Note the color and odor of ethyl alcohol. (2) Is it soluble in water? (3) Determine whether the alcohol is a good solvent for sucrose, for sodium chloride, for sulfur, for cottonseed oil.

Pour a few drops of ethyl alcohol into an evaporating dish, ignite, and (4) note the characteristics of the flame. (5) What products of combustion should you expect to be formed? (6) Test for their presence and record your result. (7) Write the equation for the combustion of alcohol.

Repeat the above experiments, using methyl alcohol.

*b. Action of iodine and sodium hydroxide on ethyl alcohol; iodoform.* Pour 2 cc. of alcohol into a test tube and add to this 5 cc. of iodine solution. Now add a solution of sodium hydroxide, *one* drop at a time (mix after the addition of each drop), until the iodine color vanishes; then warm gently and set aside for a few minutes. A yellow precipitate of *iodoform* (page 465 of text), of characteristic odor, forms. (If the amount of alcohol present is small, the iodoform may not separate, but its presence will be revealed by its odor.) This reaction serves as a good test for ethyl alcohol.

Repeat the experiment, using methyl alcohol. (8) Note the results.

*c. The properties of formaldehyde.* (9) What is an aldehyde (page 491 of text)? (10) What is the distinction between formaldehyde and formalin (page 491 of text)? (11) Note the physical properties of formalin. Add 1 or 2 drops of formalin to 6 cc. of a warm Fehling's solution as in testing for sugars (see Exercise 71). (12) Account for the results.

*d. Action of formaldehyde on silver salts.* Thoroughly clean a test tube, finally rinsing it with a little concentrated nitric acid, and then with distilled water. Now fill the tube about two thirds full of the silver nitrate solution prepared as directed under "Materials." Add to this 2 or 3 drops of formalin, quickly mix the liquids and place the tube in a beaker of warm water for eight or ten minutes; then withdraw the tube and (13) note and explain the results.

## EXERCISE 73

## SOME COAL-TAR COMPOUNDS

**Object.** To prepare some typical coal-tar compounds and to study their properties.

**Apparatus.** Test tubes; 250-cc. flask, fitted with stopper and glass tube open at both ends, as shown in Fig. 78; glass rod; 250-cc. beaker.

**Materials.** 10 cc. of benzene; 2 drops of cottonseed oil; nitric acid; sulfuric acid; 1 or 2 cc. of aniline; 5 g. of bleaching powder; 0.2 g. of naphthalene; a crystal of phenol.

NOTE. The student should first start experiment *b*; while the liquids are being heated, the remainder of the exercise can be performed.

**Caution.** *Benzene is very inflammable and must be kept away from all flames.*

*a. Benzene* ($C_6H_6$). (1) What is the distinction in composition between benzene and benzine? (2) Note the color and odor of benzene. (3) Add about 1 cc. of benzene to an equal volume of water. (4) Does it mix with water? (5) Is it heavier or lighter than water? Add 2 drops of cottonseed oil to 2 cc. of benzene. (6) Is it a good solvent for oils? Dip the end of a glass rod into the liquid and touch it to the edge of a flame for an instant. (7) Is benzene inflammable?

*b. Nitrobenzene* ($C_6H_5NO_2$). Pour 8 cc. of nitric acid into a 250-cc. flask and add (***Care***), drop by drop, with constant motion of the flask so as to mix the liquids, 10 cc. of sulfuric acid, cooling the mixture all the time by allowing cold water to run on the outside of the flask. When the mixture is cold, add, drop by drop, 6 cc. of benzene, mixing the liquids thoroughly after each addition of benzene. Now connect the flask with a cork and a glass tube (Fig. 78) and heat the mixture *very gently* (not to the boiling point). The tube acts as an air condenser to prevent the escape of the benzene. During the heating mix the liquids in the flask every few minutes by gently rotating the flask. Continue the heating for about half an hour; then disconnect the flask, cool it, and pour the contents into a 250-cc. beaker filled with cold water. Stir the mixture with a glass rod, and after the

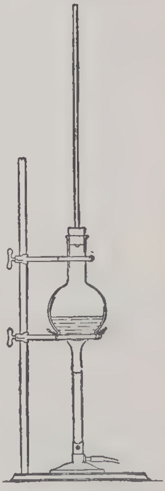


FIG. 78

oil (nitrobenzene) settles to the bottom, decant the clear liquid, leaving the oil in the beaker. Fill the beaker again with water, stir, and decant as before. In this way the acids are washed away, leaving the nitrobenzene pure.

The nitric acid reacts with the benzene to form nitrobenzene. (8) Write the equation for the reaction (page 492 of text).

The sulfuric acid assists by absorbing the water formed in the reaction. (9) Record the color of the nitrobenzene; also its odor (poisonous if too much is inhaled) and density as compared with water.

The above is the first step in the commercial production of *aniline*, which is the source material for many dyes. The nitrobenzene is converted into aniline by reduction:

$$\underset{\text{nitrobenzene}}{C_6H_5NO_2} + 3\ H_2 \longrightarrow \underset{\text{aniline}}{C_6H_5NH_2} + 2\ H_2O$$

*c. Aniline* ($C_6H_5NH_2$). (10) Note the properties (color, odor, solubility in water) of aniline. Add *one drop* of aniline to 100 cc. of water in a flask and shake the mixture. The aniline soon dissolves in the water. Place about 1 g. of bleaching powder in a beaker, add 25 cc. of water, and stir the mixture thoroughly with a glass rod; then filter and add 1 or 2 cc. of the filtrate to the aniline solution and mix thoroughly. If no change in color takes place, add more of the filtrate, 2 or 3 cc. at a time. (11) Record your results. This reaction serves as a delicate color test for aniline.

*d. Naphthalene* ($C_{10}H_8$). (12) Note the physical properties of naphthalene. Place about 0.2 g. of naphthalene in the bottom of a clean, dry test tube and apply a very gentle heat. (13) Note the results.

*e. Phenol* (*carbolic acid*) ($C_6H_5OH$). (14) Note the color, odor, and solubility in water, of phenol.

## EXERCISE 74

## VINEGAR

**Object.** To study the properties of vinegar, and especially to determine the percentage of solid matter and of acetic acid present in different samples.

**Apparatus.** Evaporating dish; beaker; ring stand and burner; 2 burettes; graduated pipette.

**Materials.** 30 cc. each of different kinds of vinegars; 1 cc. of phenolphthalein solution (R.S.); 10 cc. of sodium hydroxide solution (on desk) diluted to 100 cc. with water, or a *normal solution* (page 273 of text) of sodium hydroxide; standard oxalic acid solution containing exactly 15 g. of $H_2C_2O_4 \cdot 2\ H_2O$ to 1000 cc. (not required if a normal solution of sodium hydroxide is used).

*a. Determination of the solids in vinegar.* In this experiment different students should use different kinds of vinegar and compare results. Weigh a small evaporating dish to 0.01 g. and record the weight in the

first table below. Pour into the dish a definite volume (say 25 cc.) of vinegar. Evaporate the vinegar to complete dryness (Fig. 59); then carefully wipe the bottom of the dish with a dry cloth and again weigh, recording the weight in the table.

From your results calculate the amount of solid matter in 100 cc. of the vinegar. Pure cider vinegar should contain not less than 1.6 g. of solids in 100 cc. of vinegar (this is the limit fixed by the Federal government as well as by the statutes of many of the states). Distilled vinegar, or white vinegar, on the other hand, contains only a trace of solid matter. (1) Note the odor and taste of the solid matter obtained from the vinegar. The solids from pure cider vinegar should have an odor and taste suggestive of baked apples. The character of the solids varies according to the source of the vinegar.

Table of Results

| | |
|---|---|
| 1. Weight of evaporating dish . . . . . . . . . . . . . . . . | g. |
| 2. Weight of evaporating dish and residue . . . . . . . . . . | g. |
| 3. Weight of solids (residue) in volume of vinegar taken (Calculate) | g. |
| 4. Weight of solids in 100 cc. of vinegar (Calculate) . . . . . . | g. |

*b. Determination of the acidity of vinegar.* Any solution of sodium hydroxide of convenient *known strength* may be used. A normal solution serves the purpose well. If this is not available, the student may make up a solution of the hydroxide and determine its strength as follows: Dilute with water 10 cc. of sodium hydroxide solution on your desk to 100 cc. Measure out from a burette exactly 10 cc. of the standard oxalic acid ($H_2C_2O_4 \cdot 2\ H_2O$) solution, and add to it 2 or 3 drops of phenolphthalein solution. Fill another burette with the dilute sodium hydroxide solution and allow this to run slowly into the oxalic acid solution (see Exercise 37) until the last drop, when stirred through the acid solution, turns it a permanent pink color. Note, from the burette reading, the exact volume of the hydroxide solution used in neutralizing the acid. Now calculate the strength of the hydroxide solution. The equation for the reaction is as follows:

$$H_2C_2O_4 \cdot 2\ H_2O + 2\ NaOH \longrightarrow Na_2C_2O_4 + 4\ H_2O$$

The strength of the oxalic acid solution being known (namely, 15 g. to 1000 cc.), the strength of the sodium hydroxide solution can easily be calculated. Insert the values in the table:

Table of Results

| | |
|---|---|
| 1. Weight of oxalic acid in 10 cc. of solution . . . . . . . . . . | mg. |
| 2. Number of cubic centimeters of sodium hydroxide solution required to neutralize the oxalic acid in 10 cc. of the acid solution . . . . . . . . . . . . . . . . . . . . . . . | cc. |
| 3. Weight of sodium hydroxide in this volume . . . . . . . . | mg. |
| 4. Weight of sodium hydroxide per cubic centimeter of solution | mg. |

Having determined the strength of the sodium hydroxide solution, proceed as follows to determine the acidity of a sample of vinegar: Introduce exactly 5 cc. of vinegar into a small beaker and dilute with about 50 cc. of water. Add 2 drops of phenolphthalein and run in the solution of sodium hydroxide from a burette until the liquid in the beaker is neutral, as in Exercise 37. The equation for the reaction is as follows:

$$H \cdot C_2H_3O_2 + NaOH \longrightarrow Na \cdot C_2H_3O_2 + H_2O$$

Knowing the strength of the sodium hydroxide solution and the volume of this solution required to neutralize the acetic acid in 5 cc. of vinegar, it is easy to calculate the amount of acetic acid present in the 5 cc. of vinegar. The Federal law requires that vinegar must contain not less than 4 g. of acetic acid in 100 cc. of vinegar. (2) Insert the values for the vinegar tested in the table:

Table of Results

1. Weight of sodium hydroxide in 1 cc. of the solution used . . mg.
2. Number of cubic centimeters of hydroxide solution used to neutralize the acid in 5 cc. of vinegar . . . . . . . . . cc.
3. Weight of sodium hydroxide required to neutralize the acid in 5 cc. of vinegar (Calculate) . . . . . . . . . . . . . mg.
4. Weight of acetic acid in 5 cc. of vinegar (Calculate) . . . . mg..
5. Weight of acetic acid in 100 cc. of vinegar (Calculate) . . . g.

## EXERCISE 75

## ESTERS; OILS AND FATS; SOAP

**Object.** To study the properties of esters; to prepare soap and study its properties.

**Apparatus.** Small flask (250 cc.); evaporating dish; ring stand and burner; stirring rod; funnel and filter paper; small beaker; 4 test tubes.

**Materials.** 10 cc. of glacial acetic acid; sulfuric acid; 15 cc. of ethyl alcohol; 5 cc. of cottonseed oil; 1 g. of sodium hydroxide dissolved in 2 cc. of water; hydrochloric acid; magnesium sulfate (R.S.); calcium chloride (R.S.); 5 cc. of a saturated solution of sodium chloride.

*a. Preparation of ethyl acetate.* (1) What is an ester (page 501 of text)? Ethyl acetate is an ester derived from acetic acid by replacing an atom of hydrogen in the acid by the univalent radical *ethyl* ($C_2H_5$); it has the formula $C_2H_5 \cdot C_2H_3O_2$. It is a colorless liquid boiling at 78° and has a characteristic fragrant odor.

Pour into a small flask 10 cc. of glacial acetic acid, 5 cc. of sulfuric acid, and 3 cc. of ethyl alcohol. Mix well and heat slightly. Ethyl acetate is formed and may be recognized by its odor. Do not mistake the odor of the vapor of the alcohol for that of ethyl acetate (warm a

little ethyl alcohol in a test tube and note the difference between its odor and that of ethyl acetate). The equation for the reaction is

$$\underset{\text{acetic acid}}{H \cdot C_2H_3O_2} + C_2H_5OH \rightleftarrows \underset{\text{ethyl acetate}}{C_2H_5 \cdot C_2H_3O_2} + H_2O$$

The above reaction is reversible, as indicated, and the addition of a dehydrating agent such as sulfuric acid increases the yield of ethyl acetate. Since the ethyl acetate has a characteristic odor, easily recognized, the above reaction is often used as a test for acetic acid as well as for ethyl alcohol.

*b.* Carefully study the composition of the important compounds present in the animal and vegetable fats and oils (page 501 of text). (2) Show that these compounds are esters.

*c. Preparation of soap.* Add 10 cc. of ethyl alcohol to 5 cc. of cottonseed oil in an evaporating dish. To the resulting mixture add 1 g. of sodium hydroxide dissolved in 2 cc. of water. Evaporate carefully (use small flame and do not let the tip touch the dish), stirring the mixture constantly until the odor of alcohol can no longer be detected. (3) On the supposition that cottonseed oil is composed of olein (page 502 of text), write the equation for the reaction between cottonseed oil and sodium hydroxide. (4) What compounds remain in the dish?

*d. The reactions of soap.* Add 50 cc. of cold water to the soap prepared above, stir well for two or three minutes in order that it may dissolve, and filter. Pour about 5 cc. of the filtrate into each of four test tubes and label these 1, 2, 3, and 4. To the first, add 2 or 3 drops of hydrochloric acid; to the second, add a few drops of a solution of magnesium sulfate; to the third, a like amount of a solution of calcium chloride; and to the fourth, add a few drops of a saturated solution of sodium chloride. (5) Note carefully the results obtained in each case, and write the equations for the reactions involved, on the supposition that the soap present consists entirely of pure sodium oleate.

*e.* Recall the effect of soap in the formation of emulsions (Exercise 66). (6) What influence has this property on the cleansing action of soap?

## EXERCISE 76

## COMPOUNDS OF THE METALS

**Object.** To study some general methods for the preparation of compounds of the metals.

**Apparatus.** 6 test tubes.

**Materials.** 0.1 g. of each of the following salts, dissolved in 5 cc. of water: calcium chloride, lead nitrate, barium chloride, ferric chloride, silver nitrate (solutions of any of these on the reagent shelf may be used); ammonium carbonate solution (R.S.); 0.1 g. of potassium iodide dissolved in 5 cc. of water; hydrochloric acid.

*a. By the direct union of the elements.* Recall the formation of sulfides of copper and of iron (Exercise 53); of chlorides of copper and antimony (Exercise 33). (1) Write the equations for the reactions involved.

*b. By dissolving a metal or its hydroxide in appropriate acids.* Recall the formation of zinc sulfate (Exercise 8); of sodium chloride (Exercise 36); of copper nitrate (Exercise 43). (2) Write the equations for the reactions. (3) When a metal or its hydroxide is acted upon by an acid, what becomes of the metal?

*c. By acting upon a salt of an acid with an acid having a higher boiling point.* Recall the action of sulfuric acid upon sodium nitrate (Exercise 43); of hydrochloric acid upon iron sulfide (Exercise 54); of sulfuric acid upon fluorides (Exercise 47); of sulfuric acid on chlorides (Exercise 34); of hydrochloric acid on carbonates (Exercise 24). (4) Write the equation for each reaction and show in what respects they are all similar.

*d. By the decomposition of a compound.* Recall the action of heat upon potassium chlorate (Exercise 6); upon copper nitrate and lead nitrate (Exercise 44). (5) Write the equations for the reactions involved.

*e. By precipitation.* The following compounds are insoluble (see Appendix, "Table of Solubilities of Some Compounds of the Metals"): calcium carbonate ($CaCO_3$), lead sulfate ($PbSO_4$), barium carbonate ($BaCO_3$), ferric hydroxide ($Fe(OH)_3$), silver chloride ($AgCl$), lead iodide ($PbI_2$). Prepare a small quantity of each in a test tube. (6) State the general principle involved in each reaction and write the equation for the reaction.

## EXERCISE 77

## THE COMPOUNDS OF SODIUM

**Object.** To study the properties of some of the compounds of sodium.

**Apparatus.** Beaker; 250-cc. bottle; hard-glass test tube; platinum wire for flame test (see Exercise 65); burner; piece of cobalt glass (10 cm. square); wide-mouthed 1-liter bottle provided with a cork through which passes a right-angled glass tube reaching to the bottom of the bottle; Kipp apparatus for generating carbon dioxide; magnifying glass.

**Materials.** Litmus paper (red and blue); hydrochloric acid; 5 g. of sodium carbonate; sulfuric acid; 2 or 3 crystals of Glauber's salt; marble for generating carbon dioxide; 3 g. of sodium bicarbonate; limewater (R.S.); 35 g. of sodium chloride; 75 cc. of ammonium hydroxide.

*a.* Recall experiments with sodium (Exercise 35).

*b.* Dissolve 5 g. of sodium chloride in as little water as possible. Set the solution aside overnight or until the next laboratory period and note the crystals deposited. (1) Examine the shape of the crystals (use magnifying glass).

*c.* Dissolve 5 g. of sodium carbonate in 20 cc. of water. Test the solution with red and with blue litmus paper. (2) Note and explain the results.

Now convert the sodium carbonate present into common salt. How can you be sure that the product contains no unchanged sodium carbonate? (3) Describe the method, writing the equation for the reaction involved. Treat some of the solid salt so prepared with sulfuric acid. (4) What gas is evolved?

*d.* (5) What changes does Glauber's salt undergo when exposed to air (refer to Exercise 59)?

*e.* Fill a hard-glass test tube about one fourth full of sodium bicarbonate and heat gently. (6) Prove that carbon dioxide is evolved. (7) What liquid condenses in the colder part of the tube? (8) Write equations for the reactions by which sodium carbonate is converted into the bicarbonate, and vice versa.

*f.* Bend the end of the platinum wire (see "Apparatus") into the form of a small loop, and hold it in the outer film of the Bunsen flame until it ceases to give any color to the flame; then bring the loop into contact with a bit of sodium chloride or other compound of sodium, either in the solid state or dissolved in water, and again hold the loop in the outer film of the flame. (9) Note the color imparted to the flame. Observe the color of the flame when viewed through a piece of cobalt glass. If the glass is sufficiently thick (two pieces of the glass may be required) the yellow color of the flame disappears, so that the flame appears practically colorless. Any volatile compound of sodium will color the flame the same as sodium chloride; and this property, together with the fact that the color is not transmitted through cobalt glass, serves as a test for sodium. The test is extremely delicate, however, so that a slightly yellow color imparted to the flame by a compound may be due only to traces of some sodium compound present as an impurity.

*g. Preparation of sodium bicarbonate by the Solvay process.* Fit a wide-mouthed bottle of about 1-liter capacity with a stopper through which passes a right-angled glass tube extending to the bottom of the bottle. Place in the bottle 42 g. of sodium chloride and add to this 100 cc. of water and 75 cc. of ammonium hydroxide (sp. gr. 0.90). Stopper the bottle and gently rotate it so as to hasten the solution of the salt. When the salt is entirely dissolved, connect the glass tube with a Kipp carbon dioxide generator by means of rubber tubing 15 or 20 cm. in length. Raise the cork and tube so that the end of the tube is just above the surface of the liquid in the bottle; then pass in carbon dioxide until the bottle is entirely filled with the gas. Now close the bottle with the stopper so that the carbon dioxide entering will bubble up through the liquid in the bottom of the bottle. Leave the stopcock of the Kipp generator open so that carbon dioxide will be forced into the bottle under continuous pressure. Shake the bottle vigorously every few seconds so as to thoroughly agitate the liquid. The sodium bicarbonate formed is gradually precipitated. (10) Write the equations for the reactions involved.

NOTE. Often the precipitation of the sodium bicarbonate does not take place as rapidly as one would expect. Frequently it may be hastened by placing the bottle in ice water so as to cool the liquid. At any rate, the precipitation ought not to require more than half an hour.

## EXERCISE 78

## THE COMPOUNDS OF POTASSIUM

**Object.** To learn the test for potassium; also to prepare potassium nitrate.

**Apparatus.** Platinum wire for flame test; cobalt glass; burner; small beaker; stirring rod; funnel and filter papers to fit.

**Materials.** 0.1 g. of potassium nitrate and 0.1 g. of sodium chloride, each dissolved in a little water; 17 g. of sodium nitrate; 15 g. of potassium chloride.

*a. Test for potassium.* (1) Repeat *f*, Exercise 77, using a solution of a compound of potassium. (2) Note the appearance of the flame through a piece of cobalt glass. (3) Repeat, using a solution containing a compound of sodium as well as one of potassium. (4) How could you detect both sodium and potassium if they were present in the same solution?

*b. Preparation of potassium nitrate.* Dissolve 17 g. of sodium nitrate in 15 cc. of boiling water; also 15 g. of potassium chloride in 30 cc. of

boiling water. Mix the two solutions in a small beaker and evaporate (stirring the mixture) to about 20 cc.; then quickly filter the hot solution and set the filtrate (filtrate A) aside until cold.

When we dissolve sodium nitrate and potassium chloride in water, we are really dealing with the ions $Na^+$, $NO_3^-$, $K^+$, and $Cl^-$. Keeping in mind that either of the positively charged ions may combine with either of the negatively charged ions, we have involved in the reactions the four compounds whose solubilities at 15° and at 100° are as follows:

|  | *15°* | *100°* |  | *15°* | *100°* |
|---|---|---|---|---|---|
| Sodium nitrate . . . . | 84 | 180 | Potassium nitrate . . | 26 | 246 |
| Potassium chloride . . | 33 | 57 | Sodium chloride . . . | 36 | 40 |

Now, recalling the conditions under which ionic reactions go to completion (page 336 of text), (5) what compound should you expect to separate when the hot solutions of sodium nitrate and potassium chloride are mixed together? (6) Test the solid on the filter paper. (Examine the crystals with a magnifying glass and compare with *b*, Exercise 77, to see if your conclusion is correct. Taste the crystals.)

(7) What solid should you expect would separate from filtrate A when it is cooled? (8) Should you expect it to be pure? Examine it with a magnifying glass. (9) Can you detect crystals of sodium chloride in this solid?

Dissolve the solid in as little hot water as possible, cool the solution, and again filter off the solid. Repeat until no crystals of sodium chloride can be detected. (10) Prove the identity of the compound that separates from the cool solution.

## EXERCISE 79

## THE COMPOUNDS OF AMMONIUM

**Object.** To study the properties of the ammonium compounds.

**Apparatus.** Evaporating dish and watch-glass cover; 5 test tubes; burner; hydrogen sulfide generator (Fig. 72).

**Materials.** Ammonium hydroxide; hydrochloric acid; sodium hydroxide solution; litmus paper (red and blue); 10 g. of ferrous sulfide; 0.2 g. of ferrous sulfate dissolved in 10 cc. of water; ammonium carbonate (R.S.); barium chloride (R.S.); calcium chloride (R.S.); zinc acetate (R.S.).

*a.* Pour 10 cc. of ammonium hydroxide into an evaporating dish, neutralize with hydrochloric acid, and evaporate to dryness on a water bath (Fig. 59). (1) What compound remains in the dish? (2) Note the odor of the residue.

Introduce about one half of the residue into a test tube, add a few drops of sodium hydroxide solution, and heat gently. (3) Note the odor of the evolved gas and its action on a moist strip of red litmus paper. *All ammonium compounds evolve ammonia when heated with sodium hydroxide. This reaction serves as a good test for all ammonium compounds.*

Cover the evaporating dish containing the remainder of the residue with a watch glass and heat *gently* with a *small* flame. Note that the solid *sublimes*, that is, passes directly from the solid form into a vapor which condenses (partially) on the cold surface of the watch glass.

*b.* Saturate 5 cc. of water with hydrogen sulfide and set aside; also saturate 5 cc. of ammonium hydroxide with the hydrogen sulfide. (4) What compound (or ions) is present in each of these solutions? Add a few drops of each to separate portions of ferrous sulfate solution and (5) note the result (ferrous sulfide is soluble in strong acids). (6) In general, what metals will be precipitated as sulfides by ammonium sulfide and not by hydrogen sulfide?

*c.* Add a few drops of ammonium carbonate solution to solutions of compounds of each of the following elements: barium, calcium, zinc. (7) Write the equation for the reaction in each case.

## EXERCISE 80

## THE COMPOUNDS OF CALCIUM

**Object.** To study the properties of the compounds of calcium.

**Apparatus.** Blowpipe and charcoal; 3 test tubes; glass tubing for blowing air through solution; 250-cc. flask; platinum wire for flame test.

**Materials.** 3 g. of marble; 10 g. of commercial cyanamide; red litmus paper; calcium chloride (R.S.); strontium chloride (R.S.); barium chloride (R.S.); potassium dichromate (R.S.); ammonium sulfate (R.S.); ammonium oxalate (R.S.); ammonium carbonate (R.S.); 5 cc. of saturated solution of calcium sulfate.

*a.* Place two small pieces of marble each about as large as a pea on a piece of charcoal and heat them for five minutes in the oxidizing blowpipe flame. When cool drop the resulting solid into 10 cc. of cold water in a test tube. (1) What evidence have you of a chemical reaction? Shake the tube for about one minute; then filter and collect the filtrate. Test the filtrate with litmus paper. Account for the result. Now gently blow exhaled air through the clear filtrate. (2) Note all the changes and write the equations for the three reactions that have taken place.

*b.* (3) Write the equations for the reactions that would enable you to prepare each of the following compounds from calcium carbonate: calcium chloride, calcium sulfate, calcium cyanamide, calcium sulfite, calcium bisulfite.

*c.* Place about one tablespoonful of commercial cyanamide in a 250-cc. flask, add 50 cc. of water, and heat to boiling. Suspend a strip

of red litmus paper just inside the flask and note any change in color. (4) What do the results indicate? (5) Can you detect the odor of ammonia in the escaping steam? (6) Account for its formation. (7) What is the original source of the nitrogen present in the ammonia that is evolved? (Recall the method of making the cyanamide.)

*d.* *Action of certain reagents on solutions of compounds of calcium, strontium, and barium; tests for the ions* $Ca^{++}$, $Sr^{++}$, *and* $Ba^{++}$. Add 2 or 3 drops of a solution of potassium dichromate to 2 cc. of a solution of each of the following compounds: calcium chloride, strontium chloride, and barium chloride. Insert the results in the table below. In a similar way, on solutions of calcium chloride, strontium chloride, and barium chloride test the action of each of the following reagents: ammonium sulfate, ammonium oxalate ($(NH_4)_2C_2O_4$; see pages 500 and 559 of text), ammonium carbonate, a saturated solution of calcium sulfate. (Barium sulfate is practically insoluble in water, strontium sulfate dissolves in 10,000 parts, and calcium sulfate in 500 parts of water at ordinary temperatures.) Insert the results of your tests in the table below.

TABLE OF RESULTS

| | $Ca^{++}$ | $Sr^{++}$ | $Ba^{++}$ |
|---|---|---|---|
| Potassium dichromate . . | | | |
| Ammonium sulfate . . . . | | | |
| Ammonium oxalate . . . . | | | |
| Ammonium carbonate . . | | | |
| Calcium sulfate . . . . . . | | | |

(8) Reasoning from the results obtained above, how could you detect the barium ion in the presence of the calcium ion and the strontium ion? (9) How could you detect the strontium ion in the presence of the calcium ion?

*e.* Try the flame test for each of the following metals, as in Exercise 77 (use the solution of the chloride or nitrate of each metal): calcium, strontium, barium. Record the results below.

| METAL | COLOR OF FLAME |
|---|---|
| Calcium . . . . . . . . . . . . . . . . . . | |
| Strontium . . . . . . . . . . . . . . . . . | |
| Barium . . . . . . . . . . . . . . . . . . . | |

## EXERCISE 81

## MAGNESIUM AND ITS COMPOUNDS

**Object.** To study the properties of magnesium and of some of its compounds.

**Apparatus.** Porcelain crucible and cover; ring stand and burner; pipestem triangle; test tubes; small beaker.

**Materials.** Strips of magnesium 5 cm. long; strips of red and of blue litmus paper; 2 g. of crystals of magnesium chloride; magnesium sulfate (R.S.); ammonium chloride (R.S.); disodium phosphate (R.S.); ammonium hydroxide.

*a.* (1) Study the properties (appearance, density, etc.) of the metal. Wind a strip of magnesium ribbon into a coil and place it in a porcelain crucible. Put the cover on the crucible and apply a gentle heat. Raise the cover slightly from time to time so as to admit air. Continue until the magnesium is entirely burned, leaving a white powder. Cool and add a few drops of water. (2) Can you detect the odor of ammonia? Add 25 cc. or more of water to the solid and stir thoroughly, then (3) test with litmus. When magnesium burns in air, most of the metal combines with oxygen, but a portion of it combines with nitrogen, forming magnesium nitride ($Mg_3N_2$). When water is added, the nitride completely hydrolyzes, evolving ammonia. (4) Write the equations for all the reactions involved when magnesium is burned and the product treated with water.

*b.* Place about 2 g. of crystals of magnesium chloride in a test tube and heat it gently. (5) Is water given off easily? With blue litmus paper keep testing the drops which condense on the sides of the tube. (6) How do you account for the reaction? After most of the moisture is driven off, add water to the residue. (7) Test the resulting mixture with blue and with red litmus paper. (8) Write the equations for all the reactions involved.

*c.* Pour 3 cc. of a solution of magnesium sulfate into a test tube, add an equal volume of a solution of ammonium chloride, and then 8 or 10 drops of ammonium hydroxide. Now add to this mixture a few drops of a solution of disodium phosphate. A precipitate of magnesium ammonium phosphate ($MgNH_4PO_4$) is formed. This reaction serves as a good test for the magnesium ion $Mg^{++}$. (9) Write the equation for the reaction (see page 571 of text). The ammonium chloride is added to prevent the formation of a precipitate by the ammonium hydroxide.

## EXERCISE 82

## ZINC AND ITS COMPOUNDS

**Object.** To study the properties of zinc and of some of its compounds.

**Apparatus.** Blowpipe and charcoal; burner; 3 test tubes; beaker.

**Materials.** 0.5 g. of zinc; sulfuric acid; sodium hydroxide; ammonium sulfide (R.S.); 0.2 g. of sodium carbonate dissolved in 3 cc. of water; 2 g. of zinc sulfate; hydrochloric acid; pieces of charcoal.

*a.* Place a bit of zinc on charcoal and heat it in the tip of the flame of a blowpipe (Fig. 76). The resulting compound is deposited as a film on the charcoal. (1) Note the color of it. (2) Is its color the same when hot as when cold? (3) Write the equation for the formation of the compound.

*b.* Dissolve 0.5 g. of zinc sulfate in 20 cc. of water. Divide the solution into three parts and proceed to test the solution with the reagents named below:

*Sodium hydroxide:* Add one drop of the sodium hydroxide solution. A precipitate of zinc hydroxide is formed. (4) Note its color and write the equation for the reaction. Now continue to add sodium hydroxide (10 drops) to the precipitate formed and note the result.

*Ammonium sulfide:* Add a few drops of ammonium sulfide to another part of the zinc sulfate solution. (5) Note the color of the precipitate. (6) Write the equation for the formation of the precipitate. *Zinc is the only common metal which forms an insoluble white sulfide.* The formation of this white sulfide is therefore a good test for the zinc ion ($Zn^{++}$).

*Sodium carbonate:* Test the third portion of the zinc sulfate with a few drops of sodium carbonate solution. A basic carbonate is formed. (7) Note its color.

*c.* Devise a process for converting zinc sulfate into zinc chloride. Submit the process to your instructor for approval. When approved, prepare some of the chloride according to your process. (8) Write the equations involved.

## EXERCISE 83

## THE STORAGE BATTERY

**Object.** To demonstrate the principle of the storage battery.

**Apparatus.** Large test tube; glass rod; 50 cm. of insulated copper wire; wide-mouthed bottle; filter paper; direct current, 4 volts.

**Materials.** 2 strips of sheet lead 15 × 1 cm.; piece of sandpaper; 2 cc. of starch solution; crystal potassium iodide; sulfuric acid.

*a. The storage battery.* Prepare the apparatus illustrated in Fig. 79. The tube *A* is the large test tube that was used in the preparation of oxygen; *B* and *C* are strips of sheet lead rubbed bright with coarse sandpaper. These strips should be about 1 cm. in width, and should be bent at right angles near one end so as to rest on the rim of the test tube. In this bent end a small hole is made by driving a wire nail through the lead, and a copper wire is passed through the hole and twisted to a tight connection. When the lead plates are suspended in the tube *A*, they must be far enough apart to be separated by a glass tube *D*, so as to prevent short-circuiting. The whole cell may be slipped into a wide-mouthed bottle as a support. *E* and *F* are binding posts, terminals of two storage cells connected in series.

*b.* As a preliminary experiment obtain 1 or 2 cc. of a solution of starch (R.S.) and dissolve in it a crystal of potassium iodide. Place a clean sheet of filter paper on a clean and dry spot on the table close to the binding posts *E* and *F*, and on this pour a few drops of the solution just prepared. Attach two wires to the binding posts and bring the two free ends upon the moist starch-iodide paper about 1 cm. apart. (1) Is there any change of color where the wires touch the paper? (2) At which pole does it occur? (3) How do you explain it? (4) Will this reaction serve to detect an electric current?

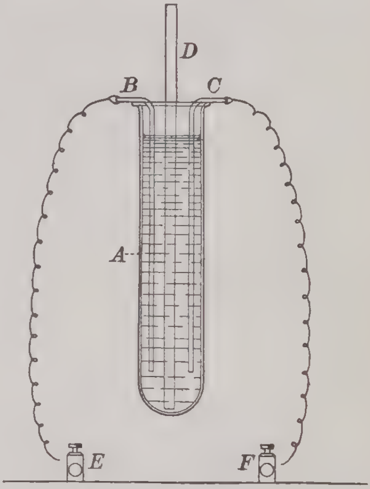


FIG. 79

*c.* Prepare an electrolyte for the cell by mixing (*Caution*) equal volumes of concentrated sulfuric acid and water, making enough of the solution to fill the tube *A* nearly full. When the solution has cooled, pour it into the tube *A* and bring the ends of the two wires connected with *B* and *C* upon the starch-iodide paper. (5) Can you detect any current? (6) Is your cell acting as a battery? Now connect the wires with the binding posts. (7) What changes in the cell do you note? (8) Which plate turns brown? (9) What is the brown substance? (10) What is formed at the other plate? (11) What gases are evolved? When one plate has become decidedly brown, disconnect the wires from *E* and *F* and bring the free ends upon the starch-iodide paper, assuring yourself that it is still moist. (12) Is iodine set free now? (13) What is the source of the current?

## EXERCISE 84

## ALUMINUM AND ITS COMPOUNDS

**Object.** To study the properties of aluminum and of some of its compounds.

**Apparatus.** 5 test tubes; blowpipe and charcoal; 2 beakers; stirring rod; ring stand and burner.

**Materials.** 0.5 g. of aluminum in the form of filings or turnings; 10 g. of aluminum powder; 1 g. of lampblack; 5 cm. of magnesium ribbon; hydrochloric acid; ammonium hydroxide; sodium hydroxide; aluminum sulfate solution (R.S.); 2 or 3 drops of cobalt nitrate solution (R.S.); 1 g. of sodium carbonate in 5 cc. of water; aluminum sulfate and potassium sulfate sufficient to make 20 g. of crystals of potassium alum (see *e*, page 138); limewater (R.S.).

*a.* Note the physical properties of aluminum. Add 5 cc. of water to 0.5 g. of the metal in a test tube; then add hydrochloric acid, a drop at a time, until the metal is dissolved. Filter (if necessary) and dilute the solution to about 10 cc. and divide it into two equal parts. To the first add ammonium hydroxide until the solution reacts alkaline. (1) Note the results (page 598 of text). To the second add sodium hydroxide, a drop at a time, until a precipitate forms. (2) Write the equation for its formation on the assumption that aluminum hydroxide is formed. Shake the tube containing the precipitated hydroxide and divide the mixture into two equal portions. To one add hydrochloric acid, (3) noting the result; to the other add sodium hydroxide solution until the precipitate dissolves. (4) Is aluminum hydroxide an acid or a base? (5) What are such compounds called?

The reaction noted above, when a solution of sodium hydroxide is added to a solution of aluminum compound, is the one commonly used for detecting the presence of the aluminum ion $Al^{+++}$. The presence of a number of other elements, however, interferes with the test.

*b.* Prepare some hydrated aluminum oxide by adding ammonium hydroxide to a solution of aluminum sulfate, and heat it on charcoal in the blowpipe flame. Moisten the residue with a drop or two of a solution of cobalt nitrate and reheat. (6) Note the result. The color formed is characteristic of aluminum oxide, and is often used as a confirmatory test for this metal. It is not conclusive however, as certain phosphates and silicates give the same color under similar treatment.

*c.* (7) What kinds of salts hydrolize most readily (pages 401–402 of text)? (8) What evidences have you that aluminum hydroxide is a weak base? (9) Is carbonic acid a strong or a weak acid (Exercise 67)? Supposing that aluminum carbonate could be prepared, (10) what reaction should you expect to take place when the carbonate is brought in contact with water? (11) What reaction should you expect to take place if a solution of sodium carbonate (or any soluble carbonate) were added to a solution of an aluminum salt such as aluminum sulfate? Carry out the experiment and see if it is in accord with your expectation.

Similarly, study the reaction between ammonium sulfide and aluminum sulfate or other soluble aluminum salt. (12) Note the results and write all the reactions involved.

*d. Aluminum nitride.* (Two or three students may work together.) Mix thoroughly 10 g. of fine aluminum powder with 1 g. of lampblack. Place the mixture in the form of a cone on a brick or iron plate. In the top of the cone introduce a piece of magnesium ribbon about 5 cm. long. Now light the magnesium ribbon. The combustion progresses safely and without explosion. When the mass is cooled, note the crystals of aluminum nitride mixed with crystals of aluminum oxide. Place some of the product in a test tube and cover it with a solution of caustic soda, heat gently, and note the odor of the gas evolved. (13) What possible use does this suggest for aluminum? (14) Write the equations for all the reactions involved.

*e. Preparation of potassium aluminum sulfate (potassium alum).* Calculate the weights of aluminum sulfate (the crystals of aluminum sulfate have the formula $Al_2(SO_4)_3 \cdot 18\,H_2O$) and of potassium sulfate required to prepare 20 g. of crystals of potassium alum; then dissolve these quantities of the two compounds separately in as little hot water as possible, mix the two solutions thoroughly, and set aside for a few days to crystallize. If a string is suspended in the liquid, the crystals will deposit on it. These may then be withdrawn and their properties studied. (15) Describe the properties of the alum.

## EXERCISE 85

## PURIFICATION AND SOFTENING OF WATER

**Object.** To study some of the properties of hard waters and the reactions involved in their purification.

**Apparatus.** 3 wide-mouthed bottles (250-cc.); graduated tube; carbon dioxide generator; 3 beakers; test tubes; ring stand and burner; burette or graduated cylinder.

**Materials.** Aluminum sulfate solution (R.S.); limewater (R.S.); pieces of marble for generating carbon dioxide; 1 g. of calcium sulfate; 2 cc. of saturated solution of sodium carbonate; 1 g. of soap dissolved in 100 cc. of distilled water; 0.5 g. of sodium sulfate.

*a.* Label the three bottles A, B, and C respectively. Nearly fill A and B with muddy water, pouring a like volume of distilled water into C. Add 5 drops of aluminum sulfate solution to A and C respectively. Mix the contents of each bottle thoroughly. Now add 10 cc. of limewater to bottle A and to bottle C. Set the bottles aside and examine at

the beginning of the next laboratory period. (1) Note and explain the results. (Bottle B is used for comparison.)

*b.* Bubble carbon dioxide into 25 cc. of limewater diluted with an equal volume of water. Note that a precipitate forms, which gradually dissolves as more of the gas is passed through. (This solution will serve as a sample of hard water.) (2) Write the equations for all the reactions involved. Add a few drops of a soap solution to 5 cc. of the hard water and note the result.

Divide the remainder of the hard water into two parts. Gradually boil one part. To the other part add a few drops of clear limewater and mix intimately. (3) Note all the results, writing the equations for the reactions.

*c.* Shake 1 g. of calcium sulfate with 10 cc. of water in a test tube for two or three minutes; filter, and add to the filtrate 2 or 3 drops of a saturated solution of sodium carbonate. (4) Account for the results.

All hard waters contain more or less calcium acid carbonate, calcium sulfate, and calcium chloride; also the corresponding compounds of magnesium. (The methods used for removing the calcium compounds likewise serve for removing the magnesium compounds.) (5) How could such waters be softened on a large scale? (6) Waters softened in this way would contain what compounds in solution?

*d. Determination of the amount of soap lost by using hard water for laundering.* Place three 250-cc. bottles on the desk. Into the first pour 100 cc. of *hard water* (preferably an average sample of the water used in your town or city); into the second pour 100 cc. of distilled (or rain) water; and into the third pour 100 cc. of distilled water containing 0.5 g. of sodium sulfate in solution. Now add to each the soap solution (see "Materials"), 1 cc. at a time, and shake the bottle *vigorously.* Continue adding the soap solution until the lather formed persists for five minutes. (7) Compare the quantities of the soap solution required in each case to produce a permanent lather. Sodium sulfate is left in waters softened by ordinary methods (page 621 of text). (8) Does the presence of this salt in the water prevent the formation of lather? (9) Calculate the weight of soap lost in the use of 10 l. of the hard water.

## EXERCISE 86

## IRON AND ITS COMPOUNDS

**Object.** To study the properties of iron and of some of its compounds.

**Apparatus.** Forceps; 2 beakers; watch glass; funnel and filter paper; flask (250-cc.); 8 test tubes; burner; platinum wire used for flame tests.

**Materials.** Watch spring from 10 to 15 cm. in length (broken watch springs can be obtained from any jeweler); 5 g. of small tacks or fine iron wire; 0.5 g. of powdered iron; sulfuric acid; nitric acid; hydrochloric acid; ammonium hydroxide; potassium ferrocyanide (R.S.); potassium ferricyanide (R.S.); ammonium thiocyanate (R.S.); sodium hydroxide; ammonium sulfide (R.S.); cobalt nitrate (R.S.); nickel nitrate (R.S.); 2 g. of borax.

*a. The tempering of steel.* Heat a piece of watch spring (from 10 to 15 cm. in length) to a white heat in a Bunsen flame. Let it cool slowly, and when cold bend it to determine whether it is brittle. Again heat to a white heat and *at once* plunge into a beaker of cold water. When cool, bend the piece as before. Reheat the piece, allow it to cool slowly, and again examine it. (1) Record the results of the experiments.

*b. Preparation of solutions of ferrous and ferric salts.* Place about 0.5 g. of iron powder in a small flask and pour over it 5 cc. of water and then 2 cc. of hydrochloric acid. Mix the contents of the flask, heat the flask gently, and set it aside in the hood for about five minutes. (2) What is formed? Now add 50 cc. of water to the flask, mix well, and filter off the undissolved iron. Nearly fill a test tube with the filtrate, add to it 4 or 5 drops of hydrochloric acid and a small piece of iron wire (or 1 or 2 tacks) and *loosely* cork the tube. Mark this Solution A. It consists of a solution of ferrous chloride. The iron wire and the hydrochloric acid generate a little hydrogen, which prevents the oxidation of the ferrous chloride to ferric chloride by the oxygen of the air. Mark the remainder of the filtrate Solution B.

*c. Reactions of ferrous and ferric salts.* To Solution B add 1 cc. of hydrochloric acid and heat it nearly to boiling; then withdraw the flame and add nitric acid, drop by drop, with constant stirring, until the solution, which is at first dark brown in color, becomes light in color (about 2 cc. of nitric acid will be required). The *ferrous chloride* in the solution is changed to *ferric chloride* by the oxygen furnished by the nitric acid. (3) Explain the reaction from the standpoint of the ionization theory. (4) Show that it is an oxidizing reaction. Now compare the action of the following reagents upon Solutions A and B (add 2 or 3 drops of the reagents to 3 cc. of the solutions in separate test tubes):

ammonium hydroxide, potassium ferrocyanide, potassium ferricyanide, ammonium thiocyanate. (5) Tabulate your results below and write the equation for the reaction in each case:

TABLE OF RESULTS

| REAGENT | FERROUS CHLORIDE (Solution A) | FERRIC CHLORIDE (Solution B) |
|---|---|---|
| Ammonium hydroxide . . . . . . . . . . . . | | |
| Potassium ferrocyanide . . . . . . . . . . . | | |
| Potassium ferricyanide . . . . . . . . . . . | | |
| Ammonium thiocyanate . . . . . . . . . . . | | |

*d. Preparation of crystallized ferrous sulfate* (*copperas*). Place 5 g. of fine iron wire or small tacks in a beaker and pour over it 15 cc. of water. Add 4 cc. of concentrated sulfuric acid and heat very gently (***Hood***) until a vigorous evolution of gas takes place; then cover the beaker with a watch glass and set it aside in the hood until near the end of the laboratory period. Then add 10 cc. of water, and heat slowly until the liquid boils, stirring the mixture constantly. Filter off any undissolved solids, collecting the filtrate in a beaker. Set the uncovered beaker containing the filtrate in your desk until the next laboratory period. If nicely formed crystals have been obtained, filter them from the mother liquor and dry them on clean filter paper. If the crystals are in the form of a solid mass, add a little water, heat until they have dissolved, and set the beaker aside for a new trial for crystallization. (6) Record the properties of the crystals. (7) Write the equation for their formation from iron.

*e.* Test separate solutions of a salt of cobalt and of nickel with a borax bead (Exercise 65); with a solution of sodium hydroxide; with ammonium sulfide. (8) Note the results, writing the equations for all the reactions involved.

*f.* Write on a piece of paper, using the cobalt nitrate solution as ink. Now heat the paper gently over a flame (invisible ink); moisten the paper with a damp cloth. (9) Note the results.

## EXERCISE 87

## DETECTION OF THE IONS OF CERTAIN METALS

**Object.** To detect the presence of certain metals when their salts are mixed together in the same solution.

**Apparatus.** Test tubes; beakers; funnel and filter papers; burner; ring stand.

**Materials.** Ferric chloride (R.S.); aluminum sulfate (R.S.); calcium chloride (R.S.); magnesium sulfate (R.S.); sodium hydroxide; hydrochloric acid; ammonium hydroxide; ammonium thiocyanate (R.S.); ammonium chloride (R.S.); ammonium carbonate (R.S.).

NOTE. It is often essential for the chemist to be able to determine just what metals are present in a mixture of the salts of the metals. The process is somewhat complicated by the fact that the reaction of any one of the metals may be interfered with by the presence of the other metals. It is necessary, therefore, to select such reactions as will obviate this complication. In carrying out such reactions the chemist finds it convenient, as a rule, to work with solutions of the compounds. It is evident, therefore, that in all such cases *we are really detecting the ions of the metals*. The principle involved in making such analyses may be illustrated in the case of detecting iron, aluminum, calcium, and magnesium in the presence of each other.

*a. Detection of ions of iron, aluminum, calcium, and magnesium* ($Fe^{+++}$, $Al^{+++}$, $Ca^{++}$, $Mg^{++}$) *in the presence of each other.* To solutions of a salt of iron (ferric) and of aluminum, respectively, add sodium hydroxide, a drop at a time, until an excess has been added. Note that the aluminum compound at first precipitated redissolves on the further addition of sodium hydroxide. To the resulting solution add hydrochloric acid until the solution becomes acid, then make it slightly alkaline with ammonium hydroxide and heat to boiling. (1) Note the result. Filter off the precipitate obtained by adding sodium hydroxide to the iron compound, dissolve a small portion of it in dilute hydrochloric acid, and add a drop of ammonium thiocyanate. (2) How can you detect ferric ions and aluminum ions in the presence of each other?

*b.* Into separate test tubes pour about 2 cc. of a solution of some compound of each of the following metals: iron, aluminum, calcium, magnesium. Add to each an equal volume of ammonium chloride solution and then a few drops of ammonium hydroxide. (3) Note the results. (4) How can you separate ions of iron and aluminum from those of calcium and magnesium?

*c.* To a solution of a salt of calcium and a solution of a salt of magnesium, add an equal volume of ammonium chloride solution, then a few drops of ammonium carbonate solution. (5) How can you verify the presence of magnesium ions (*c*, Exercise 81)?

*d.* Prepare an outline for the detection of ions of iron, aluminum, calcium, and magnesium when mixed together in the same solution. (6) Present the outline to the instructor for criticism and approval.

## EXERCISE 88

## COPPER AND ITS COMPOUNDS

**Object.** To study the properties of the compounds of copper.

**Apparatus.** 3 test tubes; beaker; ring stand and burner; hydrogen sulfide generator; evaporating dish; funnel and filter paper; stirring rod.

**Materials.** Nail; cupric sulfate solution (R. S.); sodium hydroxide; ammonium sulfide (R. S.); ammonium hydroxide; 10 cm. of copper wire; hydrochloric acid; 10 g. of copper; 5 g. of cupric sulfate; 1 g. of brass filings; nitric acid; ferrous sulfide for generating hydrogen sulfide.

*a.* (1) Recall the action of nitric acid and of sulfuric acid on copper (Exercises 43, 56); also the action of chlorine and sulfur on copper (Exercises 33, 53). Place a nail in a solution of cupric sulfate. After five minutes, (2) note and account for the result.

*b. Cuprous chloride.* Powder 5 g. of cupric sulfate and place it in a large test tube or a small beaker. Add 20 cc. of concentrated hydrochloric acid and about 10 g. of copper, preferably in a form that has a large surface. (3) What ions are now present? (4) What action should you expect metallic copper to have on the cupric ion $Cu^{++}$? Boil the mixture vigorously for about ten minutes (***Hood***) and then pour it into a beaker containing at least 200 cc. of water. The white precipitate is cuprous chloride ($CuCl$). (5) Write the equations for all the reactions. Allow the white solid to settle, and pour off most of the clear liquid. Stir the remainder, pour 5 cc. of the mixture into a test tube and add a solution of sodium hydroxide until a permanent precipitate forms. (6) What is its color? (7) What is it? (8) Write the equation.

*c.* To a *cold* solution of cupric sulfate add one half its volume of sodium hydroxide solution. Cupric hydroxide ($Cu(OH)_2$) is precipitated. (9) What color is it? Now heat to boiling. The hydroxide is decomposed into water and cupric oxide (black). (10) Write the equations for all the reactions.

*d.* Try the action of ammonium sulfide on cupric sulfate and (11) write the equation for the reaction.

*e.* Add 1 drop of ammonium hydroxide to a dilute solution of cupric sulfate; now continue to add the ammonium hydroxide, drop by drop, with constant stirring, until the precipitate which is at first formed is dissolved. (12) How does the color of the solution so obtained compare with that of the original solution? This reaction is characteristic of all compounds which give cupric ions.

*f.* (13) Recall the formation of cuprous oxide (Exercise 71).

*g.* Moisten the end of a copper wire with hydrochloric acid and hold it in the edge of a Bunsen flame. (14) Note the result.

*h. Analysis of brass.* To detect the presence of copper and zinc in brass, place 0.5 g. of brass in an evaporating dish (***Hood***) and dissolve it in as little nitric acid as possible. (15) Note the color of the solution. (16) What does it indicate? Evaporate the solution just to dryness, add 5 cc. of hydrochloric acid and 5 cc. of water, and warm gently. Transfer the clear liquid to a beaker, dilute to 100 cc. with hot water, and pass a slow current of hydrogen sulfide into the solution as long as a precipitate forms. (17) What is the precipitate? Filter, and test the filtrate with hydrogen sulfide in order to be certain that the precipitation is complete. If no more precipitate forms, evaporate the filtrate to half its volume, add a few drops of nitric acid, and again heat to boiling. When the solution is cool, add ammonium hydroxide until it is alkaline, then warm gently and set aside for a few minutes. A slight precipitate of ferric hydroxide ($Fe(OH)_3$) may form, due to traces of iron in the brass. Filter, and add ammonium sulfide to the clear filtrate (*b*, Exercise 82). (18) What is the composition of the precipitate?

## EXERCISE 89

## MERCURY AND ITS COMPOUNDS

**Object.** To study the properties of mercury and of its compounds.

**Apparatus.** Small beaker; test tubes; ring stand and burner; funnel and filter paper.

**Materials.** 2 or 3 globules of mercury; nitric acid; copper cent; 0.2 g. of mercuric nitrate crystals; hydrochloric acid; potassium iodide (R. S.); mercuric nitrate (R. S.); sodium hydroxide.

*a. Mercuric compounds.* (1) Note the physical properties of mercury. Place in a small beaker a globule of mercury as large as a grain of wheat, and add (***Hood***) just enough nitric acid to dissolve it. (2) Write the equation for the reaction on the supposition that mercuric nitrate is formed. Dilute the product with 10 cc. of water and divide it into two parts. To one part add 2 or 3 drops of sodium hydroxide solution. (3) Account for the results. In the other part place a copper cent. After a few minutes remove the coin and polish it with a piece of cloth. (4) Account for the result.

*b.* Place one or two crystals of mercuric nitrate in a dry test tube and heat gently in a small Bunsen flame, moving the bottom of the tube slowly through the flame rather than holding it steadily in the flame. (5) What gases are evolved (page 313 of text)? Continue gentle heating until colored gases cease to be evolved. (6) What remains? (7) What color is it when hot? when cold? (8) What would happen if you were to heat the product to a higher temperature? Allow the product to cool in the tube.

*c.* Dissolve the mercuric oxide obtained in *b* in as little nitric acid as possible, then add water until the test tube is one fourth full. Add to this 1 or 2 drops of hydrochloric acid. (9) Does a precipitate form?

*d.* Prepare a little mercuric iodide by precipitation. (10) What is its color at the instant of precipitation? after a few minutes? Collect it on a filter paper and allow it to dry. Scrape a little of the powder into a test tube and very carefully warm it at some distance above the flame. (11) What change in color takes place? (12) Is this change of color reversed on cooling?

*e. Mercurous compounds.* Put a small globule of mercury into a test tube and add about 3 cc. of water and 3 or 4 drops of nitric acid, warming gently and taking care that some mercury remains undissolved. After about five minutes, decant (or filter) the clear solution and add hydrochloric acid or a soluble chloride to the solution. (13) Note the results and write the equation for all the reactions involved. (14) In what respect does this reaction resemble the one employed in preparing cuprous chloride (Exercise 88, *b*)? (15) Write the essential part of each reaction as reduction in the charge of a metal ion.

## EXERCISE 90

## SILVER AND ITS COMPOUNDS

**Object.** To study the properties of the compounds of silver.

**Apparatus.** 200-cc. beaker; funnel and filter paper; blowpipe and charcoal; 4 test tubes; ring stand and burner.

**Materials.** Piece of white cotton cloth 2 or 3 cm. square; silver dime; nitric acid; hydrochloric acid; ammonium hydroxide; hot water; 2 or 3 g. of sodium carbonate; 10 cc. of silver nitrate solution (R. S.); solutions of potassium (or sodium) bromide and of potassium (or sodium) iodide (R. S.); 1 cc. of formaldehyde solution.

*a.* Place a drop of silver nitrate solution on a piece of cotton cloth and warm gently. The compound is reduced to metallic silver. (1) Can you wash the stain away?

(2) Try ammonia water. Owing to the permanence of this stain, silver nitrate is sometimes used in making indelible ink.

*b. Preparation of pure silver from a silver coin.* Place a silver dime in a small beaker and add (***Hood***) sufficient nitric acid to dissolve it. Solution may be hastened by applying a gentle heat. When the solution is complete, dilute the product with about 25 cc. of water. (3) Account for the color of the liquid. Now add a solution of hydrochloric acid until a precipitate ceases to form. On being stirred the precipitate settles to the bottom of the beaker. Carefully decant the clear supernatant liquid and add ammonium hydroxide to it until it becomes alkaline. (4) Consult *e*, Exercise 88, and account for the results. Wash the precipitate two or three times by pouring hot water over it and decanting. Finally, remove any remaining water by filtration. Mix the product with an equal bulk of sodium carbonate, transfer to a small cavity in a piece of charcoal, and heat it with a blowpipe (Fig. 76). The silver salt is gradually reduced to metallic silver, which will fuse into a globule if sufficient heat is applied.

*c.* Prepare small quantities of the chloride, the bromide, and the iodide of silver. (5) Write the equations for all the reactions. (6) Expose to the sunlight the test tubes containing the precipitates and note any changes. (7) For what are these compounds used?

*d.* Recall the formation of silver sulfide (Exercise 55).

*e.* Thoroughly clean a test tube by rinsing it with a few drops of nitric acid and then with distilled water. Pour into the tube 2 cc. of silver nitrate solution and dilute with an equal volume of water. Now add ammonium hydroxide, *a drop at a time* (shake the tube after adding each drop), until the precipitate which forms at first redissolves, leaving a clear liquid. Next add 2 drops of a solution of formaldehyde, mix thoroughly, and place the tube in a beaker of cold water. Gradually heat the water to boiling. (8) Note and account for the results.

## EXERCISE 91

## TIN AND ITS COMPOUNDS

**Object.** To study the properties of tin and of some of its compounds.

**Apparatus.** 100-cc. beaker; blowpipe and charcoal; ring stand and burner; wing-top burner.

**Materials.** 2 pieces of tin (size of a pea); hydrochloric acid; nitric acid; sodium hydroxide; mercuric chloride solution (R.S.); hydrogen sulfide solution; 5 g. stannous sulfide; 2.5 g. sulfur; 2 g. ammonium chloride.

*a.* (1) Note the physical properties of tin. (2) Heat a bit of it on charcoal with a blowpipe and note the color produced around the metal.

*b. Stannous compounds.* Dissolve some small pieces of tin in hydrochloric acid. Stannous chloride is formed. Cool, dilute to about 10 cc., and add 1 or 2 drops of the solution to 3 cc. of mercuric chloride solution. A white precipitate of mercurous chloride forms. Now add a few drops more of the stannous chloride solution and heat the mixture gently. The mercurous chloride is reduced to metallic mercury, which forms a dark-gray precipitate. (3) Write the equations for all the reactions involved. Pour a few drops into 5 cc. of a solution of hydrogen sulfide. (4) What is the color and composition of the precipitate?

*c. Stannic compounds.* To the remainder of the stannous chloride solution add 5 cc. of aqua regia and boil until there is no further odor of chlorine (*Hood*). (5) What is the compound formed? Pour a few drops of the solution into a solution of hydrogen sulfide. (6) What is the

color and composition of the precipitate? Treat a little of the solution with a few drops of sodium hydroxide. Is the precipitate soluble in excess of the reagent? (7) Write the equations for these two reactions. Add 2 or 3 drops of the solution to 3 cc. of mercuric chloride solution. (8) Is there now a precipitate? Why?

*d. Crystallized stannic sulfide.* Mix together intimately 5 g. of stannous sulfide, 2 g. of ammonium chloride, and 2.5 g. of sulfur, placing the mixture in a test tube. Clamp the tube *loosely* in a nearly horizontal position (***Hood***) and carefully heat it with a wing-top burner held in the hand, rotating the tube by twirling the rim with the fingers, and moving the burner constantly. Regulate the heat so that fumes of ammonium chloride are steadily evolved. When nearly all the fumes have escaped, allow the tube to cool, and tap the crystals out on a piece of paper. (The ammonium chloride serves to regulate the heat by its vaporization, and does not need to be considered in the reaction.) (9) To what element is sulfur similar? (10) Is the conversion of stannous sulfide into stannic sulfide a process of oxidation?

## EXERCISE 92

### LEAD AND ITS COMPOUNDS

**Object.** To study the properties of lead and of some of its compounds.

**Apparatus.** Ring stand and burner; blowpipe and charcoal; 200-cc. beaker; 5 test tubes; 100-cc. beaker; 250-cc. beaker; funnel and filter paper; glass rod.

**Materials.** 2 g. of lead; nitric acid; ammonium sulfide (R.S.); sulfuric acid; potassium chromate (R.S.); hydrochloric acid; strip of zinc; 2 g. of red lead; 10 g. of lead nitrate; 3 g. of sodium hydroxide.

*a.* Note the physical properties of the metal. Heat a small bit on charcoal with the blowpipe. (1) Is it easily melted? (2) Note and account for the coating formed on the charcoal.

*b.* Place about 1 g. of the metal in a beaker (***Hood***) and add 20 cc. of water and 5 cc. of nitric acid. Heat *gently* until the metal is dissolved. Dilute to 100 cc. and filter, if necessary, to obtain a clear solution. Call this Solution A. Now test small portions of this solution with ammonium sulfide, sulfuric acid, and potassium chromate ($K_2CrO_4$) respectively. (3) Note the color of the precipitates. Add a few drops of hydrochloric acid to a test tube one fourth full of Solution A. Lead chloride is precipitated. Heat the mixture to boiling, and if the liquid does not become clear, add just enough boiling water to dissolve the precipitate; then set it aside until cool and note the result. (4) How can you distinguish between lead chloride and silver chloride? (5) Write the equations for all the reactions involved in *b*.

In the remainder of Solution A suspend a piece of zinc. (6) Note and explain the results.

*c.* Introduce 1 g. of red lead into a 100-cc. beaker and add 5 cc. of water. Mix well and add 1 cc. of nitric acid. Stir the mixture and heat it gently. (7) Notice the change in color of the solid. Now add 25 cc. of water, stir the mixture, and filter. (8) Test the filtrate for the presence of lead. Place the filter paper and contents in a small beaker, add 2 or 3 cc. of hydrochloric acid, and stir with a glass rod so that the acid will come in contact with the solid on the paper. Heat the beaker gently. (9) What gas is evolved (color and odor)? (10) Write the equations for all the reactions.

*d.* The heavier metals form many basic salts, most of which show little tendency to crystallize, but some of which are markedly crystalline. Lead basic nitrate ($PbNO_3(OH)$) is a good example of the latter type. Prepare the salt as follows:

Dissolve 10 g. of lead nitrate in 100 cc. of hot water and divide the solution into two equal parts. Heat one to boiling and to it add a solution of sodium hydroxide (made by dissolving 3 g. of sodium hydroxide in 25 cc. of water) as long as lead hydroxide is precipitated. Be careful not to add an excess of sodium hydroxide, for this will dissolve some of the precipitate. (11) Write the equation. Wash the white solid several times by decantation. Dilute the other half of the solution of lead nitrate to about 100 cc., heat to boiling, in a large beaker, and add the pasty lead hydroxide to it. Boil until any solid remaining in suspension is distinctly crystalline, and gradually add water until all the solid has been brought into solution. (The solution may remain slightly turbid owing to lead carbonate.) If necessary, filter the solution and set it aside to crystallize. When cold, filter off the fine, needle-like crystals and dry them between layers of filter paper. (12) Write the equation for the last reaction.

## EXERCISE 93

## TESTS FOR IONS OF SILVER, LEAD, AND MERCURY

**Object.** To detect the presence of ions of silver, lead, and mercury in the presence of each other (see Note, Exercise 87).

**Apparatus.** 300-cc. beaker; ring stand and burner; stirring rod; funnel and filter paper.

**Materials.** Solutions of silver nitrate, lead nitrate, and mercurous nitrate (R. S.); blue litmus paper; potassium chromate (R. S.); sulfuric acid; ammonium hydroxide; nitric acid; hydrochloric acid.

Prepare a solution containing 0.2 g. of each of the following compounds: silver nitrate, lead nitrate, and mercurous nitrate. Dilute with water to about 200 cc. Precipitate with hydrochloric acid. (1) Write the equations for the reactions. Filter and fill the paper with boiling water three or four times, collecting the liquid as it flows from the funnel. This liquid contains the lead chloride (or its ions) which has been dissolved by the hot water. The presence of the lead ion $Pb^{++}$ may be proved by testing separate portions of the filtrate with solutions of potassium chromate and sulfuric acid respectively (see Exercise 92). (2) Write the equations for these reactions.

(3) Of what is the residue on the filter paper composed? Pour upon it 2 or 3 cc. of ammonium hydroxide and collect the liquid as it drops from the funnel. This liquid contains the silver chloride dissolved from the residue by the ammonium hydroxide. To prove its presence add nitric acid to the liquid until just acid to litmus paper. The silver chloride is precipitated.

(4) What effect did the ammonium hydroxide have upon the color of the residue on the filter paper? This change in color is due to the action of ammonium hydroxide on the mercurous chloride and serves as a test for the presence of the latter.

(5) Supposing that the original solution contained only one or two of the metals of the group, how would the absence of the remaining ones be indicated?

## EXERCISE 94

## THE COMPOUNDS OF MANGANESE

**Object.** To study the reactions of the compounds of manganese.

**Apparatus.** Test tubes; hydrogen sulfide generator; mortar and pestle; small iron dish; ring stand and burner; small beakers; burette; stirring rod.

**Materials.** Manganous chloride (R. S.); iron sulfide for generating hydrogen sulfide; ammonium sulfide (R. S.); sodium hydroxide; ammonium hydroxide; 5 g. of manganese dioxide; 5 g. of potassium hydroxide; 2.5 g. of potassium chlorate; nitric acid; sulfuric acid; 2 g. of potassium permanganate; ferrous sulfate (R. S.); 0.5 g. of pure iron wire; hydrochloric acid.

*a. Salts of manganese.* To a solution of manganous chloride add a solution of hydrogen sulfide; a solution of ammonium sulfide. Try the action of sodium hydroxide and of ammonium hydroxide. (1) Is the precipitate soluble in excess of the reagents? (2) Does it undergo any change on exposure to the air? (3) Write the equations for all the reactions involved and give the color of the precipitates formed.

*b. Manganates and permanganates.* 1. In a mortar grind 5 g. each of manganese dioxide and potassium hydroxide and 2.5 g. of potassium chlorate. Transfer the mixture to a small iron dish and heat to redness until the mass fuses and becomes deep green. The green material has the composition $K_2MnO_4$. When the mass has cooled, add cold water and dissolve as much of it as possible, decanting the solution from the residue.

2. The manganate is stable only in the presence of an excess of alkali. (4) What should you argue as to the strength of manganic acid? In water it decomposes according to the equation

$$3\,K_2MnO_4 + 4\,H_2O \rightleftarrows 2\,KMnO_4 + Mn(OH)_4 + 4\,KOH$$

Add dilute nitric acid to the solution. (5) What change in color do you note? The compound $KMnO_4$ is deep purple in color. (6) Does a precipitate form? (7) What is it? Remembering that the reaction is reversible, (8) what is the reason that nitric acid promotes the reaction from left to right? (9) How should you promote the reaction from right to left? (10) Test your conclusion.

3. (11) In the presence of sulfuric acid how does potassium permanganate act as an oxidizing agent (page 690 of text)? (12) What becomes of the manganese? (13) Are these compounds notably colored? Add 8 or 10 drops of sulfuric acid to 5 cc. of a solution of ferrous sulfate; then to this add, a drop at a time (shake the mixture after the addition of each drop), a few drops of a solution of potassium permanganate. (14) How do you account for the disappearance of the color?

Pour a little hydrochloric acid on 2 or 3 crystals of potassium permanganate and (15) account for the results.

4. Make a solution of potassium permanganate by adding about 1 g. of the salt to 100 cc. of water and fill a burette with the solution. Weigh accurately 0.5 g. of pure iron wire and dissolve it in an excess of dilute sulfuric acid. (16) What is present in the solution? Slowly add the permanganate from the burette, constantly stirring the solution. When the color of a drop added fades rather slowly, add the solution more carefully, stopping the instant a permanent pink color is produced. The continuance of the color shows that all the ferrous ions ($Fe^{++}$) have been oxidized to ferric ions ($Fe^{+++}$). (17) From the weight of the iron oxidized, calculate the weight of permanganate used up. (18) From the volume of the permanganate used, calculate the exact strength of the permanganate solution (grams in 1 liter). (19) Knowing the strength of the solution, could you determine the amount of iron present in a sample of unknown composition? This is a favorite method of analysis.

## EXERCISE 95

## THE COMPOUNDS OF CHROMIUM

**Object.** To study the reactions of the compounds of chromium.

**Apparatus.** Test tubes; iron dish; stirring rod; small beakers; ring stand; funnel; filter paper.

**Materials.** Chromium sulfate (R. S.); ammonium sulfide (R. S.); sodium carbonate (R. S.); ammonium hydroxide; sodium hydroxide; 3 g. of chromic oxide; 5 g. of potassium hydroxide; 5 g. of potassium nitrate; 10 cc. of alcohol; nitric acid; sulfuric acid; potassium hydroxide solution (R. S.); potassium chromate (R. S.); lead acetate (R. S.); potassium dichromate (R. S.); barium chloride (R. S.); hydrochloric acid; 21 g. of crystals of potassium dichromate.

*a. Salts of chromium.* Try the effect of the following reagents upon a solution of a chromium salt, (1) writing all the equations and giving the colors of the precipitates formed: ammonium sulfide, sodium carbonate, ammonium hydroxide. (2) Does sodium hydroxide produce a precipitate? (3) Is this soluble in excess of the reagent? (4) Try boiling the solution.

*b. Chromates and dichromates.* 1. In an iron dish melt equal weights of potassium hydroxide and potassium nitrate (about 5 g. of each), and gradually stir into the mixture 3 g. of chromic oxide. The nitrate is used as an oxidizing agent. (5) Explain the results (page 695 of text). Allow the fused mass to cool, then extract the soluble portion with hot water. (6) What is the color? (7) To what is it due? To 3 or 4 cc. of the solution add nitric or sulfuric acid. What change in color do you note? (8) Account for the change in color. Add potassium hydroxide to this solution. (9) Is the color change reversed? (10) Write the equation for the transformation of a chromate to a dichromate as a reversible reaction.

2. To a solution of a chromate add a solution of a soluble salt of lead. Repeat, using a salt of barium. Repeat these experiments, using a solution of a dichromate instead of a solution of a chromate. (11) Note the results and write the equations for all the reactions involved.

3. Pour 2 or 3 cc. of concentrated hydrochloric acid on a few crystals of a chromate. (12) What gas is evolved (note color and odor)?

4. Place 3 or 4 crystals of potassium dichromate in a test tube and add 2 cc. of sulfuric acid. The deep-red crystals are chromic anhydride ($CrO_3$). Pour the mixture into cold water. (13) Do the crystals dissolve? (14) What do they form?

*c. Potassium chrome alum.* When potassium dichromate in sulfuric acid solution acts as an oxidizing reagent, potassium chrome alum is formed and may be recovered in crystallized condition.

Pour 14 cc. sulfuric acid slowly (***Caution***) and with constant stirring into 45 cc. of water, and dissolve in this solution 20 g. of potassium dichromate. As a reducing agent, alcohol or starch may be used. Add 10 cc. of ethyl alcohol (or 4.5 g. starch) very slowly, stirring constantly during the energetic reaction, and being careful not to allow the temperature to rise above about 60° to prevent the hydrolysis of the chromium sulfate. As a rule no external heating is necessary, though occasionally a little may be needed to start the reaction. When the reaction has ceased, set the solution aside to crystallize. This may require some time unless the room is cold at night. Filter off the crystals and dry them on filter paper. (15) What is the color of the crystals? (16) For what are they used (page 694 of text)?

# APPENDIX

## A. TABLES OF CONSTANTS

### TENSION OF AQUEOUS VAPOR EXPRESSED IN MILLIMETERS OF MERCURY

| TEMPERATURE | PRESSURE | TEMPERATURE | PRESSURE |
|---|---|---|---|
| 12° | 10.518 | 21° | 18.650 |
| 13° | 11.231 | 22° | 19.827 |
| 14° | 11.987 | 23° | 21.068 |
| 15° | 12.788 | 24° | 22.377 |
| 16° | 13.634 | 25° | 23.756 |
| 17° | 14.530 | 26° | 25.209 |
| 18° | 15.477 | 27° | 26.739 |
| 19° | 16.477 | 28° | 28.349 |
| 20° | 17.535 | 100° | 760.00 |

### WEIGHT IN GRAMS OF 1 LITER OF VARIOUS GASES UNDER STANDARD CONDITIONS AND BOILING POINTS UNDER PRESSURE OF 760 MILLIMETERS

| NAME | WEIGHT OF 1 LITER | BOILING POINT | NAME | WEIGHT OF 1 LITER | BOILING POINT |
|---|---|---|---|---|---|
| Acetylene | 1.1621 | −83.6° | Hydrogen chloride | 1.6398 | −85.0° |
| Air | 1.2930 | | Hydrogen fluoride | | +19.4° |
| Ammonia | 0.7708 | −33.3° | Hydrogen sulfide | 1.5392 | −59.6° |
| Argon | 1.7824 | −185.7° | Methane | 0.7168 | −161.4° |
| Carbon dioxide | 1.9768 | sublimes | Nitric oxide | 1.3402 | −151.0° |
| Carbon monoxide | 1.2504 | −192.0° | Nitrogen | 1.2506 | −195.8° |
| Chlorine | 3.214 | −34.6° | Nitrous oxide | 1.9777 | −89.5° |
| Helium | 0.1785 | −268.9° | Oxygen | 1.4290 | −183.0° |
| Hydrogen | 0.08987 | −252.7° | Sulfur dioxide | 2.9266 | −10.0° |

### ELECTROCHEMICAL SERIES

1. Lithium
2. Potassium
3. Calcium
4. Sodium
5. Magnesium
6. Aluminum
7. Manganese
8. Zinc
9. Chromium
10. Iron
11. Cadmium
12. Cobalt
13. Nickel
14. Tin
15. Lead
16. Hydrogen
17. Copper
18. Arsenic, antimony, bismuth
19. Mercury
20. Silver
21. Platinum
22. Gold

## B. TABLE OF SOLUBILITIES OF SOME COMPOUNDS OF THE METALS

| | ACETATE | BROMIDE | CARBONATE | CHLORATE | CHLORIDE | CHROMATE | HYDROXIDE | IODIDE | NITRATE | OXIDE | PHOSPHATE | SILICATE (ORTHO) | SULFATE | SULFIDE |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Aluminum | W | W | | W | W | | A | W | W | A | A | A | W | A |
| Ammonium | W | W | W | W | W | W | W | W | W | | W | | W | W |
| Barium | W | W | A | W | W | A | W | W | W | A | A | A | I | W |
| Calcium | W | W | A | W | W | X | W | W | W | X | A | A | X | X |
| Cobalt | W | W | A | W | W | A | A | W | W | A | A | A | W | A |
| Copper | W | W | A | W | W | W | A | W | W | A | A | A | W | A |
| Ferric | W | W | | W | W | W | A | W | W | A | A | A | W | A |
| Ferrous | W | W | A | W | W | | A | W | W | A | A | A | W | A |
| Lead | W | X | A | W | X | A | A | X | W | A | A | A | I | A |
| Magnesium | W | W | A | W | W | W | A | W | W | A | A | A | W | A |
| Manganese | W | W | A | W | W | W | A | W | W | A | A | A | W | A |
| Mercuric | W | W | A | W | W | X | | A | W | A | A | | X | I |
| Mercurous | W | A | | W | A | A | | A | W | A | A | | X | |
| Nickel | W | W | A | W | W | A | A | W | W | A | A | A | W | A |
| Potassium | W | W | W | W | W | W | W | W | W | W | W | W | W | W |
| Silver | W | I | A | W | I | A | | I | W | A | A | | X | A |
| Sodium | W | W | W | W | W | W | W | W | W | W | W | W | W | W |
| Stannic | W | W | | | W | | A | W | | A | A | | | A |
| Stannous | W | W | | | W | A | A | W | | A | A | | W | A |
| Zinc | W | W | A | W | W | W | A | W | W | A | A | A | W | A |

W, soluble in water.
A, insoluble in water, soluble in either HCl or $HNO_3$ or in both.
I, insoluble in water and in acids.
X, slightly soluble in water and slightly or readily soluble in acids.

## C. APPARATUS AND MATERIALS REQUIRED

### 1. Reagents to be Left on Each Desk

The directions in this Manual presuppose each desk to be provided with the five reagents named below. Bottles holding 250 cc. are a convenient size. The strength of each of the acids as well as that of ammonium hydroxide is that of the ordinary commercial product. Whenever reference is made in the text to any of these five reagents it is to be understood, unless otherwise specified, that reagents of strength as given here are to be used.

Hydrochloric acid, sp. gr. 1.19. Contains from 37 to 38 per cent $HCl$; approximately 12-normal.

Nitric acid, sp. gr. 1.42. Contains 70 to 71 per cent $HNO_3$; approximately 16-normal.

Sulfuric acid, sp. gr. 1.84. Contains about 96 per cent $H_2SO_4$; approximately 36-normal.

Ammonium hydroxide, sp. gr. 0.90. Contains about 28 per cent $NH_3$ or 56 per cent $NH_4OH$; approximately 15-normal.

Sodium hydroxide, 120 g. $NaOH$ in 1000 cc. of solution; approximately 3-normal.

### 2. Apparatus to be Left on Each Desk

The following apparatus is not breakable and hence may be left on each desk for general use:

Bunsen burner with 75 cm. of rubber tubing to fit
Iron tripod (Fig. 59)
Large iron clamp like that used for holding the tube in Fig. 27
Pneumatic trough (see Appendix, D)
Ring stand and three rings of different sizes

### 3. Apparatus to be kept in Each Student's Locker

Beakers, nest of 7 (100-cc. to 700-cc.)
Blowpipe, mouth
Bottle, 5-pint acid
Bottles, wide-mouthed: 1 (60-cc.); 1 (150-cc.); 5 (250-cc.)
Burner, wing-top, for bending glass tubing (Fig. 12)
Calcium chloride drying tube, straight, 15 cm. in length (*B*, Fig. 40)
Calcium chloride drying tube, U-shaped (*C*, Fig. 40)
Charcoal, 1 piece, 8 × 3 × 2 cm. (Fig. 76)
Copper gauze, from 60 to 70 mesh, 10 × 12 cm.
Crucible, small iron
Crucible, porcelain, with lid of diameter about 3.5 cm.
Deflagrating spoon
Dish, lead, diameter about 6 cm., depth 3 cm.
Evaporating dish, diameter of top 7 cm.
File, round, about 15 cm. in length
File, triangular, about 15 cm. in length
Filters, 25, diameter about 11 cm.
Flasks, two 250-cc., one 500-cc.
Forceps, small, brass or iron
Funnel, diameter about 6.5 cm.
Funnel tube, external diameter of tube 6 mm.
Glass rod, diameter 3 mm., 1 piece 15 cm. in length
Glass rod, diameter 3 mm., 1 piece 10 cm. in length
Glass tubing, 400 g., soft, external diameter 6 mm., walls 1 mm. thick

Glass tubing, hard, internal diameter 12 mm., 1 piece 25 cm. in length (*A*, Fig. 53)
Glass tubing, hard, internal diameter 6 mm., 1 piece 30 cm. in length (*C*, Fig. 74)
Medicine dropper (Fig. 56)
Mortar, diameter about 8 cm., and pestle (both of porcelain)
Pipestem triangle, for holding porcelain crucible (Fig. 30)
Pipette, 5-cc., graduated in cubic centimeters
Platinum wire, small (No. 22), 8 cm. long, for flame tests
Retort, glass-stoppered, 150-cc. (Fig. 66)
Rubber tubing, internal diameter 6 mm., 1 piece 50 cm. in length
Rubber tubing (soft), pure gum, internal diameter 5 mm., 1 piece 60 cm. in length, for connections, etc.
Sand bath (small iron dish), 10 cm.
Screw clamp (*A*, Fig. 28)
Splints, wooden (ordinary cigar-lighters), 100
Sponge
Stopper, rubber, one-holed, to fit hard-glass test tube
Stopper, rubber, two-holed, to fit 250-cc. wide-mouthed bottle
Stopper, rubber, two-holed, to fit 60-cc. wide-mouthed bottle
Stopper, rubber, two-holed, to fit 1000-cc. narrow-mouthed bottle
Stoppers (two), rubber, one-holed, to fit drying tube (Fig. 40)
Test tube, graduated, 30-cc., about 20 cm. long, with 0.5-cc. graduations
Test tube, hard glass, 15 cm. in length, diameter about 1.8 cm.
Test tubes, 12, length 12 cm., diameter about 1.7 cm.
Test-tube brush
Test-tube rack
Towel
Watch glass, diameter about 8 cm.
Window glass, 4 pieces, 10 cm. square
Wire gauze, 2 pieces, 12 cm. square

### 4. General Apparatus for Ten Students

The apparatus listed below is general in character. It should be kept in the storeroom and drawn out by students when needed.

4 sets of apparatus for testing the conductivity of solutions (Fig. 64) (see Appendix, D)
1 balance, weighing from 0.5 g. to 500 g., with accompanying weights
2 balances, sensitive to 1 cg. and made to carry a load of 100 g.
1 barometer
4 burettes, 50-cc., graduated in 0.1 cc. (Fig. 63)
5 pieces of cobalt glass 10 cm. square (for flame tests)
4 condensers, Liebig (Fig. 39), with rubber tubing and large clamp
2 sets of cork-borers (6 in a set)
2 gross of corks, best grades, sizes 7, 8, 9, 10, and 12
1 cylinder, glass, about 30 cm. in length and from 4 to 5 cm. in width
1 cylinder, graduated, 500-cc.
1 cylinder, graduated, 200-cc.
1 distilling apparatus for preparing distilled water
2 Kipp generators
1 magnet, horseshoe, small
1 magnifying glass, small
1 microscope, eyepiece 1 inch, objective $\frac{2}{3}$ and $\frac{1}{6}$.
5 thermometers, graduated from $-10°$ to $+150°$ C.
2 tubes, graduated in cubic centimeters, 50 cm. in length and about 2 cm. in width
2 sets of weights, from 1 cg. to 50 g., in a covered wooden box

## 5. Chemicals on Reagent Shelf (for Use of All Students)

These reagents are designated in the exercises by the abbreviation "R.S." printed after the name of the reagent.

The bottles containing solutions should be glass-stoppered except those containing potassium hydroxide and sodium hydroxide, which should have hard rubber stoppers. Gummed letters of the alphabet, of different sizes, may be obtained at little cost from any stationer, and these may be used in making the labels for the bottles. If the class is small, bottles holding 250 cc. will ordinarily serve; if the class is large, it is better to use bottles holding at least 500 cc. A few of the reagents, such as limewater, are used so extensively that it is better to use a 1000-cc. bottle. Distilled water must be used in making all solutions. All solutions of salts are molar solutions unless special directions are given. The numbers in parentheses are the molar weights of the crystallized solid to give 1 liter of solution.

Acetic acid, ordinary commercial 36 per cent; approximately 6-normal
Aluminum sulfate (668)
Ammonium carbonate (96 g. of the solid dissolved in 300 cc. of water and 40 cc. of ammonium hydroxide (sp. gr. 0.90), and the solution diluted to 1000 cc. with water)
Ammonium chloride (54)
Ammonium molybdate solution (see Appendix, D)
Ammonium oxalate (124)
Ammonium sulfate (132)
Ammonium sulfide solution (168)
Ammonium thiocyanate (76)
Barium chloride (244)
Borax (solid)
Calcium chloride (111 anhydrous; 219 crystallized)
Calcium sulfate (saturated solution)
Carbon tetrachloride
Chlorine water (water saturated with chlorine; freshly prepared as needed)
Chloroform
Chromium sulfate (662)
Cobalt nitrate ($\frac{1}{5}$ molar, 58)
Cupric sulfate (250)
Disodium phosphate ($\frac{1}{2}$ molar, 179)
Ferric chloride (270)
Ferric sulfate ($\frac{1}{2}$ molar, 200)
Ferrous sulfate (278) (About 5 cc. of hydrochloric acid and a few tacks added to each 100 cc. of solution to prevent oxidation)
Formalin
Gold chloride (3 g. in 500 cc. water)
Hydrochloric acid (exactly normal solution)
Hydrogen sulfide solution (saturated; freshly prepared)
Iodine solution (prepared by dissolving 2 g. of iodine and 10 g. of potassium iodide in 100 cc. of water)
Lead acetate (379)
Lead nitrate (331)
Limewater (saturated solution of calcium hydroxide)
Litmus solution (enough litmus (cubes) to impart a deep color, dissolved in water and filtered)
Magnesium sulfate (246)
Manganese chloride (198)

Manganese dioxide (solid)
Mercuric chloride ($\frac{1}{5}$ molar, 54)
Mercuric nitrate ($\frac{1}{2}$ molar, 234)
Mercurous nitrate ($\frac{1}{5}$ molar, 60; a little mercury added)
Methyl orange (0.3 g. dissolved in 1 liter of water)
Nickel nitrate ($\frac{1}{5}$ molar, 58)
Nitric acid (fuming)
Oxalic acid (standard solution for Exercise 74; prepared by dissolving exactly 15 g c.p. oxalic acid ($H_2C_2O_4 \cdot 2\,H_2O$) in 1000 cc. of water)
Phenolphthalein (1 g. dissolved in 200 cc. of alcohol)
Potassium antimonyl tartrate (tartar emetic) ($\frac{1}{10}$ molar, 33)
Potassium bromide (119)
Potassium chlorate (solid)
Potassium chromate (194)
Potassium dichromate ($\frac{1}{3}$ molar, 98)
Potassium ferricyanide ($\frac{1}{2}$ molar, 164)
Potassium ferrocyanide ($\frac{1}{2}$ molar, 211)
Potassium hydroxide (3-normal; 168.312 g. in 1000 cc.)
Potassium iodide ($\frac{1}{2}$ molar, 83)
Sand (very fine)
Silver nitrate ($\frac{1}{4}$ molar, 42)
Soap (1 per cent solution)
Sodium carbonate (286)
Sodium chloride (solid)
Sodium chloride (saturated solution)
Sodium hydroxide (exactly normal solution; 40.005 g. in 1000 cc.)
Sodium peroxide (solid)
Starch solution (see Appendix, D)
Strontium chloride (267)
Sulfur (solid)
Urea (10 g. per 1000 cc.)
Water glass (sp. gr. 1.1)
Zinc (mossy)
Zinc acetate ($\frac{1}{2}$ molar, 119)
Zinc sulfate (287)

## 6. Chemicals required for a Class of Ten

The terms in parentheses after the names of the chemicals refer to the grade of materials to be purchased. The abbreviation "c.p." signifies "chemically pure."

This list does not include substances always easily obtained, such as sugar, salt, lard, lime, clay, coal, cotton, iron wire, broken watch springs, calico strips, lead (pieces cut from lead pipe), sandpaper, soap, kerosene, gasoline, candles, cardboard, starch, chalk, cloth, milk, vinegar, and butter.

Dealers in chemicals usually have supplies ready for sale in standard packages. It is more economical to purchase standard packages than to specify very small or unusual quantities. From the size of the class and the stock on hand, a list of approximate quantities should be made up and the dealer should be asked for an estimate of costs based on the next larger commercial packages.

Suitable containers should be provided in the laboratory in which students should be directed to put all residues of expensive materials, such as ammonium molybdate and compounds of silver and mercury. From time to time these residues should be worked up into standard materials.

| | Approximate Quantities |
|---|---|
| Acid, acetic (99.6 per cent) (glacial) | 100 g. |
| Acid, acetic (36 per cent) (commercial) | 500 g. |
| Acid, benzoic | 10 g. |
| Acid, formic (50 per cent) | 300 g. |
| Acid, hydrochloric (sp. gr. 1.2) (c. p.) | 2.5 kg. |
| Acid, nitric (sp. gr. 1.4) (c. p.) | 3 kg. |
| Acid, oxalic (c. p.) | 50 g. |
| Acid, pyrogallic | 50 g. |
| Acid, sulfuric (sp. gr. 1.84) (c. p.) | 4 kg. |
| Acid, tannic (commercial) | 100 g. |
| Agar | 50 g. |
| Alcohol, ethyl (95 per cent) | 1 kg. |
| Alcohol, methyl (methanol) | 250 g. |
| Alum (ammonium) (pure) | 100 g. |
| Aluminum (turnings or filings) (pure) | 50 g. |
| Aluminum (powder) | 100 g. |
| Aluminum sulfate (pure, crystals) | 500 g. |
| Ammonium carbonate (pure) | 100 g. |
| Ammonium chloride (pure) | 200 g. |
| Ammonium hydroxide (sp. gr. 0.90) (c. p.) | 1.5 kg. |
| Ammonium molybdate (see Appendix, D) | 500 g. |
| Ammonium nitrate (pure) | 100 g. |
| Ammonium oxalate | 100 g. |
| Ammonium sulfate (commercial) | 200 g. |
| Ammonium sulfide solution | 500 g. |
| Ammonium thiocyanate | 100 g. |
| Aniline | 50 g. |
| Antimony | 30 g. |
| Antimony trichloride | 25 g. |
| Antimony oxide | 10 g. |
| Arsenic | 30 g. |
| Arsenic trioxide (arsenious oxide) (commercial) | 30 g. |
| Auric chloride | 1 g. |
| Barium chloride (c. p.) | 100 g. |
| Barium hydroxide | 200 g. |
| Benzene | 200 g. |
| Bismuth | 30 g. |
| Bismuth chloride | 25 g. |
| Bleaching powder | 500 g. |
| Boneblack | 250 g. |
| Borax (commercial) | 100 g. |
| Boric acid (pure) | 100 g. |
| Brass filings | 10 g. |
| Bromine | 200 g. |
| Cadmium | 25 g. |
| Cadmium chloride (c. p.) | 25 g. |
| Calcium carbide | 100 g. |
| Calcium carbonate (precipitated) | 500 g. |
| Calcium chloride (fused or granular) | 1 kg. |
| Calcium fluoride (fluorite) | 200 g. |
| Calcium hydroxide (hydrated lime) | 500 g. |
| Calcium sulfate (plaster of Paris) | 1 kg. |
| Carbon disulfide (commercial) | 250 g. |
| Carbon tetrachloride (commercial) | 500 g. |
| Charcoal, pieces (8 × 3 × 2 cm.) | 10 |
| Charcoal, powdered | 100 g. |

| | |
|---|---|
| Charcoal, small pieces | 100 g. |
| Chloroform | 250 g. |
| Chromium oxide | 50 g. |
| Chromium sulfate | 100 g. |
| Cobalt nitrate (pure) | 25 g. |
| Copper (turnings or scrap) | 300 g. |
| Copper foil (thin) | 100 g. |
| Cottonseed oil | 200 g. |
| Cupric bromide | 25 g. |
| Cupric nitrate (pure) | 25 g. |
| Cupric oxide (pure, finely powdered) | 200 g. |
| Cupric sulfate crystals (c. p.) | 400 g. |
| Cupromercuric iodide (see Appendix, D) | 5 g. |
| Cuprous oxide | 60 g. |
| Cyanamide (commercial) | 1 kg. |
| Dyes (Alizarin paste (20 per cent), Bismarck brown, Congo red, gallein, fuchsine, malachite green, methyl violet, thymol blue) | 5 g. of each |
| Ether, sulfuric | 100 g. |
| Formalin | 100 g. |
| Gelatin | 50 g. |
| Glucose (sirup) or Karo corn sirup | 500 g. |
| Gypsum (crystals) | 200 g. |
| Hydrogen peroxide | 200 g. |
| Iodine | 10 g. |
| Iron chloride (ferric) (c. p.) | 100 g. |
| Iron powder (iron reduced by alcohol) | 150 g. |
| Iron sulfate (ferric) | 200 g. |
| Iron sulfate (ferrous) | 200 g. |
| Iron sulfide (ferrous) | 1 kg. |
| Iron wire (picture-frame wire), No. 0 | 25 yd. |
| Lampblack | 15 g. |
| Lead (pure, granular) | 50 g. |
| Lead, red | 25 g. |
| Lead acetate (sugar of lead) (powdered) | 200 g |
| Lead monoxide (commercial) (litharge) | 150 g. |
| Lead nitrate (pure) | 350 g. |
| Lead strips (15 × 1 cm.) | 2 |
| Litmus cubes | 50 g. |
| Litmus paper — 100 strips red, 100 strips blue | 1 tube of each |
| Magnesium carbonate (powdered) | 100 g. |
| Magnesium chloride | 50 g. |
| Magnesium ribbon | 50 g. |
| Magnesium sulfate (Epsom salts) | 250 g. |
| Manganese chloride | 100 g. |
| Manganese dioxide (commercial) | 1 kg. |
| Marble (pieces size of a walnut) | 3 kg. |
| Mercuric chloride (corrosive sublimate) | 50 g. |
| Mercuric nitrate (c. p.) | 200 g. |
| Mercuric oxide | 25 g. |
| Mercurous nitrate (c. p.) | 100 g. |
| Mercury | 50 g. |
| Methyl orange | 10 g. |
| Napthalene (moth balls) | 100 g. |
| Nickel nitrate | 25 g. |
| Paraffin | 100 g. |
| Phenol crystals | 25 g. |
| Phenolphthalein | 25 g. |

| | |
|---|---|
| Phosphorus | 15 g. |
| Phosphorus trichloride | 50 g. |
| Potassium antimonyl tartrate (tartar emetic) | 75 g. |
| Potassium bitartrate (cream of tartar) | 100 g. |
| Potassium bromide (granular) (pure) | 100 g. |
| Potassium carbonate (c. p.) | 50 g. |
| Potassium chlorate (small crystals) | 500 g. |
| Potassium chloride (c. p.) | 350 g. |
| Potassium chromate (pure) (crystals) | 100 g. |
| Potassium chromium sulfate (chrome alum) | 100 g. |
| Potassium dichromate (pure) | 500 g. |
| Potassium ferricyanide (c. p.) | 100 g. |
| Potassium ferrocyanide (c. p.) | 100 g. |
| Potassium hydroxide (sticks) (electrolytic) | 350 g. |
| Potassium iodide (pure) | 50 g. |
| Potassium nitrate (pure) | 200 g. |
| Potassium perchlorate | 60 g. |
| Potassium permanganate (pure) | 50 g. |
| Potassium sulfate (pure) | 200 g. |
| Silver nitrate | 25 g. |
| Silver oxide (purchased or prepared according to Appendix, D) | 10 g. |
| Silver powder (see Appendix, D) | 1 g. |
| Soda lime (granular) | 500 g. |
| Sodium | 25 g. |
| Sodium acetate (fused) | 200 g. |
| Sodium ammonium phosphate | 50 g. |
| Sodium benzoate (pure) | 25 g. |
| Sodium bicarbonate (baking soda) | 100 g. |
| Sodium bromide | 50 g. |
| Sodium carbonate (pure) (anhydrous) | 500 g. |
| Sodium hydrogen phosphate (disodium phosphate) (c. p.) | 100 g. |
| Sodium hydroxide (sticks) (purified by alcohol) | 1 kg. |
| Sodium iodide | 50 g. |
| Sodium nitrate (pure) | 300 g. |
| Sodium nitrite | 100 g. |
| Sodium peroxide | 200 g. |
| Sodium potassium tartrate (Rochelle salts) (powdered) | 500 g. |
| Sodium silicate solution (water glass) | 1 kg. |
| Sodium sulfate (crystals) | 200 g. |
| Sodium sulfite | 50 g. |
| Sodium thiosulfate | 150 g. |
| Stannous sulfide | 50 g. |
| Strontium nitrate | 25 g. |
| Sulfur (brimstone) | 400 g. |
| Sulfur, powdered | 250 g. |
| Tin (mossy) | 75 g. |
| Urea | 50 g. |
| Zinc (mossy, arsenic-free) | 1 kg. |
| Zinc (sheet) | 200 g. |
| Zinc acetate (c. p.) | 25 g. |
| Zinc sulfate (crystals) | 300 g. |

## D. SPECIAL APPARATUS AND MATERIALS

### 1. Apparatus

*The balance.* It is very desirable that the balance used in quantitative exercises should weigh with accuracy to 1 mg. Such a balance, of simple type, may be bought from any one of a number of firms. Very fair results may be secured with a balance weighing to 0.01g. A good example of such a balance is the Trœmner No. 13. The type known as the Harvard balance has the advantage that the weights are not easily lost.

*Pneumatic trough.* A convenient size for a pneumatic trough is 30 cm. × 20 cm. × 15 cm. It should be made of galvanized sheet iron and should be painted to protect it from rust. A supply of these troughs can be made by a local tinsmith. Of course any kind of rather deep pan or earthern crock will serve most uses.

*Conductivity apparatus.* The apparatus for measuring the conducting power of solutions (Fig. 64) may be obtained from dealers in apparatus or may be made by anyone who wires houses for electric lights. The drawing supplies the needed information.

The strips of lead used in Exercise 92 should be cut from thin sheet lead such as can be had in any good tinshop. They should be about 1 cm. wide and 15 cm. long.

### 2. Materials

*Standard solutions of acids.* It is a tedious task to make solutions of acids of exactly standard strength. The following directions will serve to make the approximate strengths indicated:

278 cc. sulfuric acid (sp. gr. 1.84) will make 5 liters of 2-normal acid.
629 cc. nitric acid (sp. gr. 1.42) will make 5 liters of 2-normal acid.
809 cc. hydrochloric acid (sp. gr. 1.19) will make 5 liters of 2-normal acid.
1590 cc. 36 per cent acetic acid will make 5 liters of 2-normal acid.
663 cc. ammonium hydroxide (sp. gr. 0.90) will make 5 liters of 2-normal alkali.

*Starch solution.* In a mortar, rub 4 or 5 g. of starch and a little water to a smooth paste. Add this paste, a few drops at a time, to 1 liter of boiling water. Add also about 10 g. of zinc chloride to act as a preservative. Mix thoroughly and set aside. Use the clear supernatant liquid.

*Preparation of silver powder.* Prepare a suitable quantity of silver chloride by precipitation from a solution of silver nitrate with dilute hydrochloric acid. Wash the chloride thoroughly by decantation and dissolve it in ammonium hydroxide. To the ammonia solution add somewhat more than the calculated cuprous chloride in the form of finely ground solid. Reduction of the silver chloride occurs at once, and the silver is deposited as a fine gray powder. Filter off the silver powder with a filter pump, wash it thoroughly with water till all traces of copper have disappeared, and dry it in an air bath at 110°–120°.

*Preparation of silver oxide* ($Ag_2O$). Dissolve 100 g. of silver nitrate in about 1 liter of distilled water. Also dissolve 25 g. of sodium hydroxide (purified by alcohol) in about 200 cc. of distilled water. Now add the sodium hydroxide solution slowly to the silver nitrate solution, stirring the mixture constantly during the addition. After about four fifths of the hydroxide solution has been added, allow the precipitated silver oxide to settle, and then continue to add, a few drops at a time, the sodium hydroxide solution until it no longer forms a precipitate when brought in contact with the clear supernatant liquid. Allow the precipitate to settle and pour off the supernatant liquid. Add about 500 cc. of distilled water to the precipitate, stir thoroughly, and again decant. Repeat the operation five times in order that the silver oxide may be freed from all impurities. Filter off the precipitated silver oxide, using a Buchner filter and suction flask. (An ordinary filter will do, but the process is not so rapid.) Again wash the precipitate on the filter paper three or four times; then remove the precipitate and dry it on a steam plate. Finally powder the solid and heat it in an air bath to 150° for about two hours. Care must be taken not to allow the temperature of the air bath to rise much above 150°, as the silver oxide begins to decompose at about 200°. When the product has thus been thoroughly dried, place it in a clean, dry bottle tightly corked.

*Sodium hypobromite for urea test.* Make two solutions as follows: (A) Dissolve 125 g. sodium bromide and 125 g. (41 cc.) bromine in water to make 1 liter of solution.

(B) Dissolve 225 g. sodium hydroxide in water to make 1 liter solution. Preserve the two solutions in separate (rubber-stoppered) bottles until needed. For use, take 100 cc. (A), 100 cc. (B), and dilute to 500 cc. of solution.

*Ammonium molybdate.* The solution of ammonium molybdate is prepared as follows: Dilute 200 cc. of concentrated nitric acid (sp. gr. 1.4) with 300 cc. of water. Dissolve 75 g. of ammonium molybdate in 500 cc. of water and slowly pour this solution into the dilute nitric acid. A precipitate of molybdic acid will at first precipitate but will afterwards dissolve.

*Cupromercuric iodide* ($Cu_2HgI_4$). Treat a solution of 6.8 g. of mercuric chloride with a solution of 8.3 g. of potassium iodide. Wash the precipitate by decantation and dissolve it in a solution of 8.3 g. of potassium iodide in 50 cc. of water. Treat this solution with a concentrated solution of 12 g. of copper sulfate and conduct into the solution a current of sulfur dioxide for the reduction of the cupric salt. The deep-red precipitate is cupromercuric iodide. Wash it with water and dry at a moderate temperature. Yield, about 20 g. If desired, small portions of this preparation may be sealed in glass tubes and handed out in this condition. Other compounds with a change of color at the transition point are silver mercuric iodide ($Ag_2HgI_4$), transition at 45°–50°; silver iodide (AgI), transition at 146°; mercuric iodide ($HgI_2$), transition at 127° but slow to reverse.

## E. FIRST-AID TREATMENT FOR BURNS, CUTS, WOUNDS, SCALDS, COLLAPSE, AND POISONING

Every laboratory should be supplied with the materials necessary for the treatment of burns, cuts, wounds, scalds, collapse, and poisoning. Such distressing accidents, when *immediately and intelligently* treated, usually give little or no trouble; but if left to take care of themselves, infection may occur in some cases and serious results follow. *In case of a severe injury the student, after preliminary treatment in the laboratory, should be sent to a physician.*

The materials required for the treatment of injuries should be kept in the laboratory in a tight cabinet or cupboard located in a conspicuous and easily accessible place, which may be emphasized by placing above the compartment a white cardboard on which a red cross is printed.

## First-Aid Materials

Plain sterile gauze bandage: 6 rolls, 1 in. by 10 yd.; 6 rolls, 2 in. by 10 yd.
Adhesive plaster for holding gauze in place: 2 rolls, 0.5 in. by 10 yd.
Picric acid gauze
Sodium bicarbonate solution, 5 per cent
Boric acid solution, 5 per cent
Yellow or white vaseline
Carron oil (equal volumes of lime water and linseed or cottonseed oil)
Iodine solution, 3.5 per cent (U. S. P. tincture diluted with an equal volume of water or 95 per cent alcohol)
Benzene
Cottonseed oil
Universal antidote. This consists of an intimate mixture of the following composition:

| | |
|---|---|
| Pulverized charcoal | Two parts |
| Magnesium oxide (magnesia) | One part |
| Tannic acid | One part |

## Burns

Note. *Remember that immediate treatment is of the greatest importance. Especially is this true in the case of burns resulting from contact with acids and alkalies.*

1. *From acids.* Wash the injury with large volumes of water, apply gauze saturated with the sodium bicarbonate solution, and then bandage.
2. *From alkalies.* Wash the injury with large volumes of water, apply a gauze saturated with boric acid solution, and then bandage.
3. *From flames and hot objects.* Apply a gauze saturated with carron oil and then bandage. If tissues have been destroyed, bind over the wound a pad of moist picric acid gauze. Keep the pad moist and change the dressing daily. If blisters have been formed, puncture as described under "Scalds."
4. *In the eyes. Immediately* wash the eyes with large volumes of water, then wash with sodium bicarbonate solution if the injury has been due to acids, or with boric acid solution in case of burns due to alkalies. Complete the treatment in either case by adding a drop of olive, castor, or cottonseed oil to act as a soothing agent.

## Cuts

Remove any dirt or grease from the wound and surrounding skin by washing with gauze saturated with benzene. A rather free bleeding at first will help to prevent infection. Finally wash the wound with a piece of gauze saturated with the iodine solution; then bandage so as to prevent infection.

## Scalds

Blisters should be drained by puncturing at two places with a flame-sterilized needle and then forcing out the liquid by the pressure of the fingers. If the injury has been produced by either acids or alkalies, it should be neutralized with the appropriate solution.

## COLLAPSE

In cases of collapse, ammonia or smelling salts may be inhaled or cold packs may be applied to the head. Brisk rubbing of the chest and extremities may be sufficient to restore one who has fainted.

## POISONING

1. Call a physician immediately or convey the patient to the hospital without delay.
2. *Universal antidote* (first aid). When a case of poisoning is suspected, the patient should immediately take the *universal antidote* described under First-Aid Materials. The dose should be one heaping teaspoonful of the mixture well stirred in a small glass of warm water.

4. Potassium nitrate is more soluble than $NaCl$: 4 $NaCl$ to 1 $KNO_3$

6. Yes

7. Dissolve sugar in water
oils or grease in carbon tetrachloride
By melting. two nothing

8. Cooler

oap Book

## TABLE OF THE CHEMICAL ELEMENTS, INCLUDING THEIR SYMBOLS, THEIR ATOMIC NUMBERS, AND THEIR ATOMIC WEIGHTS

| NAME | SYMBOL | ATOMIC NUMBER | ATOMIC WEIGHT | NAME | SYMBOL | ATOMIC NUMBER | ATOMIC WEIGHT |
|---|---|---|---|---|---|---|---|
| Actinium | Ac | 89 | — | Mercury | Hg | 80 | 200.61 |
| Aluminum | Al | 13 | 26.97 | Molybdenum | Mo | 42 | 96.0 |
| Antimony | Sb | 51 | 121.77 | Neodymium | Nd | 60 | 144.27 |
| Argon | A | 18 | 39.94 | Neon | Ne | 10 | 20.183 |
| Arsenic | As | 33 | 74.96 | Nickel | Ni | 28 | 58.69 |
| Barium | Ba | 56 | 137.36 | Nitrogen | N | 7 | 14.008 |
| Beryllium | Be | 4 | 9.02 | Osmium | Os | 76 | 190.8 |
| Bismuth | Bi | 83 | 209.00 | Oxygen | O | 8 | 16.000 |
| Boron | B | 5 | 10.82 | Palladium | Pd | 46 | 106.7 |
| Bromine | Br | 35 | 79.916 | Phosphorus | P | 15 | 31.02 |
| Cadmium | Cd | 48 | 112.41 | Platinum | Pt | 78 | 195.23 |
| Calcium | Ca | 20 | 40.07 | Polonium | Po | 84 | 210.(?) |
| Carbon | C | 6 | 12.000 | Potassium | K | 19 | 39.10 |
| Cerium | Ce | 58 | 140.13 | Praseodymium | Pr | 59 | 140.92 |
| Cesium | Cs | 55 | 132.81 | Protoactinium | Pa | 91 | — |
| Chlorine | Cl | 17 | 35.457 | Radium | Ra | 88 | 225.97 |
| Chromium | Cr | 24 | 52.01 | Radon | Rn | 86 | 222. |
| Cobalt | Co | 27 | 58.94 | Rhenium | Re | 75 | 188.7 |
| Columbium | Cb | 41 | 93.1 | Rhodium | Rh | 45 | 102.91 |
| Copper | Cu | 29 | 63.57 | Rubidium | Rb | 37 | 85.44 |
| Dysprosium | Dy | 66 | 162.46 | Ruthenium | Ru | 44 | 101.7 |
| Erbium | Er | 68 | 167.64 | Samarium | Sm | 62 | 150.43 |
| Europium | Eu | 63 | 152.0 | Scandium | Sc | 21 | 45.10 |
| Fluorine | F | 9 | 19.00 | Selenium | Se | 34 | 79.2 |
| Gadolinium | Gd | 64 | 157.26 | Silicon | Si | 14 | 28.06 |
| Gallium | Ga | 31 | 69.72 | Silver | Ag | 47 | 107.880 |
| Germanium | Ge | 32 | 72.60 | Sodium | Na | 11 | 22.997 |
| Gold | Au | 79 | 197.2 | Strontium | Sr | 38 | 87.63 |
| Hafnium | Hf | 72 | 178.6 | Sulfur | S | 16 | 32.06 |
| Helium | He | 2 | 4.002 | Tantalum | Ta | 73 | 181.5 |
| Holmium | Ho | 67 | 163.5 | Tellurium | Te | 52 | 127.5 |
| Hydrogen | H | 1 | 1.008 | Terbium | Tb | 65 | 159.2 |
| Illinium | Il | 61 | — | Thallium | Tl | 81 | 204.39 |
| Indium | In | 49 | 114.8 | Thorium | Th | 90 | 232.12 |
| Iodine | I | 53 | 126.932 | Thulium | Tm | 69 | 169.4 |
| Iridium | Ir | 77 | 193.1 | Tin | Sn | 50 | 118.70 |
| Iron | Fe | 26 | 55.84 | Titanium | Ti | 22 | 47.90 |
| Krypton | Kr | 36 | 82.9 | Tungsten | W | 74 | 184.0 |
| Lanthanum | La | 57 | 138.90 | Uranium | U | 92 | 238.14 |
| Lead | Pb | 82 | 207.22 | Vanadium | V | 23 | 50.96 |
| Lithium | Li | 3 | 6.940 | Xenon | Xe | 54 | 130.2 |
| Lutecium | Lu | 71 | 175.0 | Ytterbium | Yb | 70 | 173.6 |
| Magnesium | Mg | 12 | 24.32 | Yttrium | Y | 39 | 88.92 |
| Manganese | Mn | 25 | 54.93 | Zinc | Zn | 30 | 65.38 |
| Masurium (?) | Ma | 43 | — | Zirconium | Zr | 40 | 91.22 |